

LABORATORY
MANUAL
OF
Medical Chemistry
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LABORATORY MANUAL OF MEDICAL CHEMISTRY

A LABORATORY MANUAL
OF
MEDICAL CHEMISTRY

CONTAINING A SYSTEMATIC
COURSE OF EXPERIMENTS

IN

*Laboratory Manipulation and Chemical Action, the Non-Metallic
Elements and the Medicinal Metals, Quantitative Processes
applied to Sanitary Water Analysis, Medicinal Organic
Compounds, Proteids, Digestion, Blood,
Milk, Urinalysis and Toxicology*

BY

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PREFACE.

This volume is the outgrowth of several years of laboratory instruction given medical students. The exercises have been used in various forms by seven classes since the organization of the Medical Department. They have been gradually shaped by the peculiar needs of students of medicine, and by personal visitation and careful study of the laboratories and methods of instruction in nearly all of the leading medical schools of the United States during the years 1893-6.

Since there is not known to the author a small volume of modern, progressive laboratory exercises, covering the various branches of medical chemistry here presented, it has been decided to put these exercises into permanent form for the use of our own students.

The aim has been to give such a scope to the earlier exercises as to quickly furnish the student a broad chemical foundation necessary to an appreciative understanding of the complex field of medical chemistry; to escape, on the one hand, the danger of teaching pure theoretical chemistry, unadapted to the needs of a physician; and, on the other, the more dangerous error of mechanically drill-

ing tests and operations which the student has not the chemical knowledge to remember nor intelligently apply.

The work has been kept within such limits that it can be completed in two sessions by students working two laboratory periods each week. The more difficult and elaborate experiments have been reserved for the lecture table.

The author trusts that the errors, so easily creeping into the first edition of a work covering so large a field, will be charitably regarded.

FORT WORTH UNIVERSITY,

I. C. C.

FORT WORTH, TEXAS, *October 15, 1897.*

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EXERCISES
IN
LABORATORY MANIPULATION
AND
CHEMICAL ACTION

PRACTICAL MEASUREMENTS.

1. Comparison of English and Metric Length Units.

1. Measure with the rule the length of several objects in both systems.
2. Familiarize the length of a centimeter and decimeter.
3. Note the number of centimeters in an inch.
4. Measure the area of a book cover in square inches and square centimeters. Express each of these areas in decimal parts of a square yard and a square meter, respectively. Note how much more difficult it is to reduce the English than the metric measures.

2. Comparison of English and Metric Volume Units.

1. Using graduates, determine the capacity of the different sized test tubes, beakers and evaporating dishes in ounces, drachms and cubic centimeters (c.c.).
2. Note the number of cubic centimeters in a drachm.
3. Express the volume of your beaker in parts of a gallon and parts of a liter. Note the advantage of the metric system.

3. Comparison of English and Metric Weight Units.

1. Weigh out 5 grains of calomel, 5 grains of antifebrin and $\frac{1}{60}$ of a grain of atropine hydrosulphate.
2. Note the number of grains required to balance a gram weight.

3. Weigh out 1 gram (g.) and 10 grams of salt. Remember the quantity by noting how large a coin each quantity might just be piled upon.

4. Comparison of English and Metric Temperature Units.

1. Examine the Fahrenheit and Centigrade thermometers, noting the comparative freezing and boiling points.

2. Note the number of degrees F. corresponding to 1° C.

5. **Reduction of English and Metric Units.** By the following rules,¹ reduce the prescriptions and temperatures written on the blackboard to corresponding units in the other system.

RULE I. *To Reduce Metric to English Weights and Measures.* Multiply the quantity in grams or cubic centimeters by 10, add $\frac{1}{2}$, and the result is grains or minims (nearly).

RULE II. *To Reduce English to Metric Weights and Measures.* Divide the quantity in grains or minims by 10, subtract $\frac{1}{3}$, and the result is grams or cubic centimeters (nearly).

RULE III. *To Reduce Fahrenheit to Centigrade Temperatures.* Subtract 32, and multiply by $\frac{5}{9}$.

RULE IV. *To Reduce Centigrade to Fahrenheit Temperatures.* Multiply by $\frac{9}{5}$, and add 32.

NOTE 1. Rules I and II are merely approximate, but near enough for physicians' use, and are easily remembered and mentally applied.

CONSTRUCTION OF APPARATUS.

6. Construction of a Stirring Rod. Select a solid or hollow glass tube 8 inches long. Hold the ends in a Bunsen flame until the ends are melted together and neatly rounded. When cool, cap one end with a small piece of rubber tubing, to protect a glass beaker while stirring.

7. Construction of a Dropper. Select a piece of glass tubing 8 inches long, that will snugly fit the rubber nipple. Heat this tube at the center, with constant rotation over the Bunsen flame. When hot, draw it out an inch and a half. With a file, scratch it in the center and break it. If it has been evenly drawn, two droppers may be made. Lay one-half aside for experiment 8 (*b*). Take the other and, heating the larger end very hot, stamp it quickly on a cool metal surface, thus forming a ring to hold the rubber cap.

8. Construction of a Generator. Select a 7-inch test tube and fit it with a 1-hole and a 2-hole rubber stopper. This apparatus is referred to throughout the laboratory course as a "*generator*." It is used for a variety of purposes, and may have two kinds of fixtures, referred to as (*a*) a delivery tube, and (*b*) an ignition jet. A thistle tube added is often useful. Examine these generators on the demonstrator's desk. Fit one up, and keep it constantly ready for use.

(*a*) *Construction of a Delivery Tube.* Select a glass tube a foot long. One-third the distance from one end heat it evenly, with constant rotation, over a fish-tail burner. When hot, remove the tube from the flame and bend it into syphon

form. The bend should be uniform, with no "buckle" in the glass.

(b) *Construction of an Ignition Jet.* Round the ends of the tube saved in experiment 7, in the flame. When cool, insert it in the 2-hole cork for an ignition jet.

(c) *Construction of a Thistle Tube.* Select a glass tube 8 inches long. Heat one end very hot until closed with a bead of white-hot glass. Remove it from the flame and gently blow a bulb as large as a small marble. Next heat, with a blowpipe, a spot on the very top of the bulb until white-hot. Remove it from the flame and blow a quick, strong blast. The top will be blown out. Clean the edges from fine glass and round them by heating in the flame. This makes a small but serviceable thistle tube. Insert it in the cork by the side of the ignition jet, as in the "model" generator.

9. To Mend a Test Tube. Heat the lower portion of a bottomless test tube, and when hot draw it together with a glass rod and draw away superfluous glass. Reheat the end, remove from the flame, and gently blow it into shape. The bottom should be round and quite thin, to stand heating. To mend a large tube or generator, it will be necessary to use the bellows and blast lamp.

CHEMICAL MANIPULATION.

10. Solution. Dissolve about 1 g. of common salt, NaCl, in a test tube of water, and preserve the solution.

11. Evaporation. Put a few drops of the above solution in a porcelain evaporating dish. Place this on a piece of

wire gauze or asbestos board and heat it over a non-luminous Bunsen flame until dry. Water and salt can thus be separated by heat.

12. Precipitation. Take half the remaining salt solution. Add nearly an equal quantity of silver nitrate, AgNO_3 . A white precipitate falls. Set the tube aside for the next experiment.

13. Filtration. Prepare a funnel, filter paper, ring-stand, and beaker, as on the demonstration desk. Shake the tube prepared in the last experiment, and pour the contents down a glass stirring-rod into the filter. Reject the solution caught in the beaker below. Save the precipitate (ppt.).

14. Decantation. Precipitate the remainder of the salt solution left in experiment 12, by silver nitrate, AgNO_3 . Heat the tube, and set it aside until the precipitate settles, then pour off or decant the solution, leaving the ppt.

15. Reduction. Bore a shallow hole in a piece of charcoal. In this place all the white ppt. formed in the above experiments. Turn down the Bunsen burner to a 2-inch luminous flame. Rest the blowpipe tip on the top of the burner. Blow gently and continuously from the cheeks, and heat the precipitate with the fine blowpipe flame until minute beads of metallic silver appear.

16. Distillation. In a generator place a salt solution colored with indigo. Boil it, and condense the steam by letting the delivery tube dip in a test tube cooled by being immersed in a beaker of water. Taste the distillate, and note that both the color and salt have been removed.

17. Dialysis. Make a small dialyzer by tying tightly a fresh, thin sausage skin over the mouth of a bottomless test

tube. In this place a mixture of salt, starch and water. Set this, skin down, in a beaker of water. After some time take some of the water from the beaker and add silver nitrate. A white precipitate indicates salt, which has penetrated the membrane. The water in the beaker is not colored blue by tincture of iodine. The milky liquid in the dialyzer turns blue with iodine, showing that the starch has not passed through the membrane.

18. Electrolysis. Prepare a solution of copper sulphate, CuSO_4 , in a beaker. Attach a clean silver coin to the negative electrode of the dynamo or battery. Suspend the coin and the positive wire in the liquid without their touching. Examine the coin after a few moments.

LABORATORY QUESTIONS.

1. Is the force which holds salt in solution a weak or strong one?
2. How could you separate salt and water?
3. What is a filtrate? What is a precipitate?
4. How may a precipitate be washed on the filter paper?
5. How may a precipitate be washed by decantation?
6. What is a distillate?
7. Can silver be dissolved in water?
8. Where did the copper come from which plated the coin?
9. Why was the coin hung on the negative electrode?
10. What class of bodies passes through animal membranes?

CHEMICAL CHANGE.

19. A Mechanical Mixture. Make a mixture in any proportion of fine iron filings and flowers of sulphur. Grind them together in a mortar. This is a mixture, because the iron and sulphur can be separated by mechanical means.

(a) From a small portion of the mixture remove the iron by the use of a magnet. The sulphur will be left.

(b) To another small portion add carbon disulphide, CS_2 . Shake it up. The sulphur will be dissolved. The iron will remain. Pour the solution into an evaporating dish and set it aside. The sulphur will be left in crystals when the CS_2 evaporates.

20. A Chemical Compound. Several students together weigh out 56 g. of iron filings and 32 g. of flowers of sulphur. Mix and grind them thoroughly in a mortar. Take an inch of this mixture in a test tube. Heat it until the red-hot glow diffuses itself throughout the mass. Cool, break the test tube, and grind the mass in a clean mortar. This substance is now a chemical compound, FeS , because the iron and sulphur cannot be separated by mechanical, but only by chemical means.

(a) From a portion of the compound try to separate the iron by a magnet. It is all slightly magnetic, but no iron can be separated and sulphur left.

(b) With another portion of the compound try to dissolve out the sulphur by carbon disulphide, CS_2 . If the work has been carefully performed, no sulphur will be found on evaporating the CS_2 .

Thus iron and sulphur can unite only in definite proportion by weight, as 56 parts of iron with 32 parts of sulphur. Any extra iron or sulphur will remain as a mixture.

EVIDENCES OF A CHEMICAL CHANGE.

21. An Elevation of Temperature. Powder separately a pinch of sugar and a pinch of potassium chlorate, KClO_3 . Mix them on an iron plate, and touch the mixture cautiously with a drop of sulphuric acid, H_2SO_4 . Brilliant deflagration ensues. In general, every chemical combination is attended by an elevation of temperature.

22. The Formation of a Precipitate. Make a solution of mercuric chloride, HgCl_2 , by dissolving a little of the salt in water. Do the same with a little tin chloride, SnCl_2 . The solutions should be clear. Mix the two, and a white ppt. falls, turning black, and sometimes by heating and stirring mercury will separate. The formation of a ppt. from two clear solutions indicates a chemical change, resulting in the formation or liberation of a new insoluble substance.

23. The Evolution of a Gas. Cover marble dust with water in a test tube and add a little dilute hydrochloric acid, HCl . A gas is given off. The evolution of a gas from two solid or liquid bodies indicates a chemical change, resulting in the formation of a new gaseous substance.

24. A Change in Electrical Condition. Fill a beaker one-third full of water and add 20 drops of sulphuric acid, H_2SO_4 . Connect a zinc and a copper wire to a delicate galvanometer. Dip the wires, without touching each other, into the beaker of diluted acid. The acid forms a chemical compound with the zinc, and a current of electricity flows through the gal-

vanometer from the copper to the zinc, as indicated by the movement of the needle. In general, it is supposed that every chemical change is attended by a change of electrical condition.

25. A Change of Color. Touch a crystal of sugar of lead, lead acetate, $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$, with a drop of a potassium iodide, KI, solution. A new yellow-colored compound is formed. A change of color is *often* an evidence of a chemical change, resulting in the formation of a new and differently colored substance.

26. A Change of Reaction. Place in a beaker a little dilute hydrochloric acid, HCl. Add a small piece of blue litmus paper. The paper turns red and the solution tastes sour. Add some sodium hydroxide, NaOH, solution, with constant stirring until the paper turns faintly blue. The solution loses its acidity, and tastes salty. A change in the reaction of a solution toward litmus paper indicates a chemical change, resulting in the union of an acid and an alkali to form a new compound.

SOME CAUSES INDUCING A CHEMICAL CHANGE.

27. Heat. Place a few grains of sugar in the bottom of a test tube. Heat gently. Note the carbon remaining and the water on the sides of the tube. Heat in general favors chemical change by increasing the molecular motion, destroying old and encouraging new combinations.

28. Light. Make a solution of salt, NaCl, and add some silver nitrate solution, AgNO_3 . A white ppt. falls. Filter the solution. Place the filter paper, with its white silver chloride, in the sun. It soon turns bluish black, from a chemical change due to the energy of the sunlight.

29. Electricity. Wet a piece of filter paper in starch mucilage test solution (see 57, Note 1). Lay this on a metal plate. Firmly press the wire from the negative electrode of the dynamo or battery on the plate, and with the wire from the positive electrode write slowly upon the paper. A blue line is traced. The color is due to a chemical change, caused by electric energy.

30. Mechanical Energy. Notice that dry quicklime, CaO , and dry ammonium chloride, NH_4Cl , have no odor. Mix a little of each very loosely in a mortar. They have no odor. Press and rub them firmly with a pestle. An odor of ammonia is evolved, and the mixture grows moist. The intimate union caused by pressure has induced a chemical change.

31. Solution. Mix in a beaker some dry powdered sodium bicarbonate, NaHCO_3 , and powdered tartaric acid, $\text{H}_2(\text{H}_4\text{C}_4\text{O}_6)$. No action ensues. Add water. Gas is evolved. In general, solution favors chemical action by separating atoms and molecules, and bringing them into intimate association.

LABORATORY QUESTIONS.

1. What is meant by an element; an atom; a molecule; valence, and bond?
2. What is a mechanical mixture? A chemical compound?
3. Name several mechanical mixtures and chemical compounds in nature.
4. What is meant by the indestructibility of matter?
5. What is the law of definite proportion? How is it proved?
6. What is the law of multiple proportion?
7. In the manufacture of FeS (20), what was the most striking evidence of a chemical change?
8. When ammonium nitrate and sodium sulphate are dissolved the solution becomes cold. What evidence is there of a chemical change?
9. Is ice cream frozen by a chemical change?

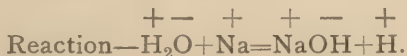
10. From what force is most of the energy of the world obtained?
11. What power unites chemical elements? How does it seem related to electricity or magnetism.
12. When a solid compound is dissolved, are the atoms separated?
13. Is solution a chemical change?
14. What is meant by atomic and molecular weight?
15. What is the molecular weight of one of the most complex substances known, provided its exact formula be $C_{600}H_{960}N_{154}FeS_3O_{179}$ (oxyhaemoglobin)?
16. What is a graphic formula?

LABORATORY EXERCISES
IN THE
MORE IMPORTANT
NON-METALLIC ELEMENTS

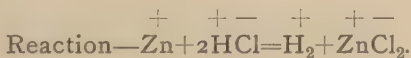
HYDROGEN.

USEFUL DATA: Atomic Symbol, H; Molecular Formula, H_2 ; Valence, 1; Atomic Weight, 1; Electro-positive; 1 liter at $0^\circ C.$ and 760 m.m. weighs .0896 g.

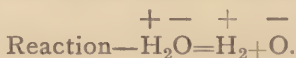
32. Preparation from Water by Sodium. Fill a test tube one-third full of water. Drop into it a small piece of metallic sodium, Na. Quickly apply a flame to the mouth of the tube. H burns with a pale blue flame. When the action ceases, test the remaining liquid by the touch, taste and action on red litmus paper. Evaporate the solution. Examine the salt remaining, which is sodium hydrate, NaOH, or solid caustic soda.



33. Preparation from Hydrochloric Acid by Zinc. In a generator put 10 g. of granulated zinc, Zn, covered with water. Add a little hydrochloric acid, HCl. Collect several test tubes of H by the displacement of water, as illustrated on the demonstration desk. Light the H in one test tube. Transfer the H from one test tube to another, remembering that it is lighter than air. Prove its presence in the last tube by igniting it. After the action in the generator entirely ceases, pick out the Zn remaining, and return it to the container. Filter the solution. Evaporate it to a small bulk, and set aside to cool. Crystals of zinc chloride, $ZnCl_2$, will separate.



34. Preparation by Electrolysis. Fill a beaker with water and add 15 drops of sulphuric acid, H_2SO_4 . In this invert a test tube filled with water. Keep the two electrodes from the dynamo or battery separated, and insert them in the beaker. Collect the tube half full of the gas arising from the negative electrode, from which the greater number of bubbles arises. Cover the tube with the thumb. Invert it, apply a flame to the gas, and prove that it burns like H.



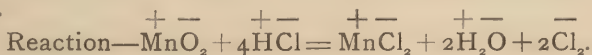
LABORATORY QUESTIONS.

1. Name a number of substances which contain H.
2. Can H be prepared from any substance which does not contain it?
3. How many atoms of H in two molecules?
4. What were the qualities of the solution remaining in 32?
5. How much will 120 c.c. of H weigh under standard conditions?
6. How do the experiments prove H electro-positive?
7. From the experiments, which would one judge more strongly electro-positive, H or Zn?
8. How much H is contained in a pound of water, H_2O ? In a pound of HCl ?
9. Is H soluble in water?
10. How does the H flame differ from that of illuminating gas?
11. Does H in the experiments act like a metal?
12. Were all these experiments examples of chemical change? Why?
13. Were any materials used in these experiments destroyed?
14. Would Zn and H be likely to form a stable chemical compound? Why?

CHLORINE.

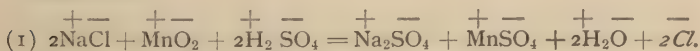
USEFUL DATA: Atomic Symbol, Cl; Molecular Formula, Cl₂; Valence, 1; Atomic Weight, 35.5; Electro-negative; 1 liter at 0° C. and 760 m.m. weighs 3.173 g.

35. Preparation from Hydrochloric Acid by Manganese Dioxide. Place a little manganese dioxide, MnO₂, in a generator. Add hydrochloric acid, HCl, and warm gently. Note the color, odor and specific gravity of the gas¹ as it rises in the tube. Wash the material quickly into the sewer.

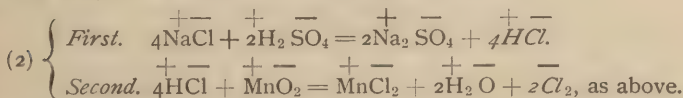


36. Preparation from a Chloride. In a generator mix two parts of sodium chloride, NaCl, and one part of manganese dioxide. Add some sulphuric acid, H₂SO₄, and gently warm. Pass the yellow gas into half a test tube of water, thus forming chlorine water. In a part of this a scale of gold leaf will dissolve. Set the rest away in the sun, and notice after some hours the change in color, odor, taste and action on litmus paper. It turns to hydrochloric acid, HCl.

Reaction—



Or it may be written in two stages, thus:



NOTE 1. Care should be taken not to allow any considerable amount of Cl to escape in the laboratory. When inhaled it produces great irritation.

37. Preparation from Bleaching Powder by an Acid.

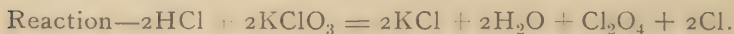
Place some bleaching powder in a generator. Add dilute acid. Hold a little damp litmus paper, colored cloth, red ink writing or other organic colors in the gas. The colors are bleached. The color does not return when they are moistened with ammonia water, NH_4OH .

38. Preparation from Hydrochloric Acid by Electrolysis.

Put some hydrochloric acid into a test tube. Insert the carbon electrodes of the dynamo or battery, keeping them separate. Note that, as before, H is liberated from the negative electrode. Cl is liberated from the positive electrode, and dissolves in the water, forming yellow chlorine water. Note the odor of chlorine.



39. Preparation of Chlorine Water. Place a few crystals of potassium chlorate, KClO_3 , in a test tube. Add 3 c.c. of hydrochloric acid. Warm until Cl escapes freely, then fill the tube with water. This is the most convenient way to prepare a little fresh chlorine water, so frequently needed in testing. The salt and acid present do not interfere with its use.

**COMPOUNDS OF CHLORINE AND HYDROGEN.**

Hydrochloric Acid (Muriatic Acid), HCl .

40. Preparation by Synthesis. Fill two test tubes, one with H and the other with Cl, from the generators in the hood. Join their mouths. Invert several times, to mix. Cautiously open the mixture to a flame. After combination, shake the gas in the two tubes with 5 c.c. of water.

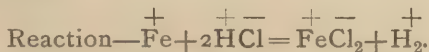
Test this by taste and blue litmus and set it aside, and later prove the presence of HCl by 43.



41. Preparation from a Chloride by Sulphuric Acid. In a generator cover 5 g. of sodium chloride with sulphuric acid, H_2SO_4 . Warm, and cautiously note the odor. Hold moist blue litmus paper in the gas until it changes to red. Pass the gas through 10 c.c. of water. A solution of HCl forms, which is the muriatic acid of commerce and medicine. Try to boil the HCl from the solution.



42. Formation of Chlorides. In an evaporating dish put 15 c.c. of HCl. Add a gram of iron filings and gently warm for ten minutes. Fill the dish with water. Filter, evaporate the clear filtrate to a small bulk, and set aside. After a few moments fine crystals of green ferrous chloride, FeCl_2 , will form.



43. The Silver Nitrate Test for Chlorides. Silver nitrate, AgNO_3 , added to a solution of chlorides, precipitates white silver chloride, AgCl , which is soluble in ammonia but insoluble in nitric acid.

44. Detonation of Chlorates. Place in an iron mortar two small crystals of potassium chlorate, KClO_3 . Add a few grains of dry sugar or flowers of sulphur. Very gently rub them to a powder and strike the mixture with the pestle. A sharp explosion will occur.

45. The Ignition Test for Chlorates. A solution of potassium chlorate, KClO_3 , yields no precipitate with silver nitrate,

AgNO_3 . Ignite a crystal of KClO_3 on a piece of glass or porcelain¹. Cool, and dissolve the residue. The chlorate has turned to a chloride, and its solution yields the test for chlorides with silver nitrate, 43.

LABORATORY QUESTIONS.

1. Name several substances that contain chlorine.
 2. Are all substances containing chlorine chlorides?
 3. When is a solution neutral to litmus?
 4. How many substances can you find that turn red litmus blue?
- Blue litmus red?
5. How do the experiments prove chlorine electro-negative?
 6. Write the reaction which chlorine water undergoes in the sun?
 7. What two compounds form the pure hydrochloric acid of commerce?
 8. Why is HCl called "spirit of salt"? What are muriates?
 9. How much Cl in one pound of salt?
 10. How much NaCl is required to manufacture one pound of HCl ?
 11. Outline a good method for bleaching white cloth?
 12. How would one proceed to disinfect a room with chlorine?
 13. If Cl unites with water, to what substance is the bleaching due?
 14. Write the symbols for K , Cu , Au , Sn , Hg , Al , Mn , Ca , Ba and NH_4 chlorides.
 15. How might all these chlorides be formed?
 16. What chloride was formed in the preparation of H ?
 17. What chlorides were formed in the preparation of Cl ?
 18. What results when chlorates are triturated with organic substances?
 19. To what acid is sodium chloride related?
 20. To what acid is potassium chlorate related?

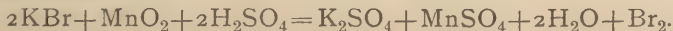
NOTE 1. See the reaction occurring here, 55, where it will be noted that oxygen is at the same time liberated.

BROMINE.

USEFUL DATA: Atomic Symbol, Br; Molecular Formula, Br₂; Valence, 1; Atomic Weight, 80; Electro-negative.

46. Preparation from a Bromide. In a generator, mix three well-powdered crystals of potassium bromide, KBr, with an equal quantity of manganese dioxide, MnO₂. Add sulphuric acid, H₂SO₄, and warm gently. Note the color, odor, specific gravity and bleaching qualities of the gas as it rises in the tube. If possible, pass the gas through water, forming bromine water. Set this aside for future use.

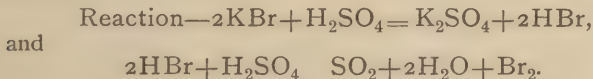
Reaction—



COMPOUNDS OF BROMINE AND HYDROGEN.

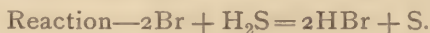
Hydrobromic Acid, HBr.

47. Preparation from a Bromide by Sulphuric Acid. Place a crystal of KBr in an evaporating dish. Add a few drops of sulphuric acid, H₂SO₄, and warm. Note the white vapors of HBr mixed with some yellowish brown vapors of free bromine.



48. Preparation from a Bromide by Hydrogen Sulphide. Take a test tube of bromine water, containing a globule of carbon disulphide, CS₂. Pass through this hydrogen sulphide, H₂S, from the generator in the hood. Shake well,

and pour off the clear solution containing HBr. Preserve this for the following tests.



49. Tests for Bromides.

1. *The Chlorine Test.* To a solution of bromides add a globule of carbon disulphide, CS_2 (chloroform may be used). Next add some chlorine water and shake. Free Br is liberated, and tinges the CS_2 yellow to brownish red, according to the amount present.

2. *The Silver Nitrate Test.* AgNO_3 ppts. in solutions of bromides yellowish white, AgBr . Insoluble in nitric acid, HNO_3 , and sparingly soluble in dilute ammonium hydroxide, NH_4OH .

3. *The Starch-bromide Test.* When starch water is added to a solution of bromides and Br liberated by a few drops of chlorine water, yellow starch bromide is formed.

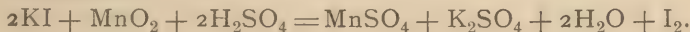
IODINE.

USEFUL DATA: Atomic Symbol, I; Molecular Formular, I_2 ; Valence, 1; Atomic Weight, 127; Electro-negative.

50. **Physical Properties.** Note the characteristics of metallic iodine. Heat a crystal in a dry test tube. Notice the color of the vapor, and the minute crystals sublimed on the side of the tube. Try to dissolve the crystals in water. Next try alcohol. Next try carbon disulphide or chloroform, and note the differences.

50 1-2. Preparation from an Iodide. In a generator mix a crystal of KI with manganese dioxide, MnO_2 . Add sulphuric acid and warm gently. Note the violet vapors of iodine as they rise in the tube.

Reaction—

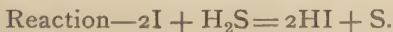


COMPOUNDS OF IODINE AND HYDROGEN.

Hydriodic Acid, HI.

51. Preparation from Iodine. Prepare a test tube containing a globule of CS_2 , a crystal of I and some water. From the generator in the hood pass hydrogen sulphide gas, H_2S , through the mixture. Hydriodic acid remains in solution.

Save this solution for the following tests.



52. Tests for Iodides.

1. *The Chlorine Test.* To a solution of iodides add a globule of carbon disulphide, CS_2 , or chloroform. Next add chlorine water, and shake. Free I is liberated and colors the globule violet to black, depending upon the amount present.

2. *The Silver Nitrate Test.* AgNO_3 , added to a solution of iodides, ppts. light yellow, AgI ; insoluble in nitric acid, HNO_3 , and sparingly soluble in dilute ammonium hydroxide, NH_4OH .

3. *The Starch-iodide Test.* When starch water is added to a solution of iodides, and free iodine liberated by a few drops of chlorine water, blue starch iodide is formed.

FLUORINE.

USEFUL DATA: Atomic Symbol, F; Valence, 1; Atomic Weight, 19; Electro-negative.

COMPOUNDS OF FLUORINE AND HYDROGEN.

Hydrofluoric Acid, HF.

53. Preparation from a Fluoride. First cover one side of a glass plate with an even coating of paraffin, and with a sharp point scratch a name or design through the wax. Next moisten 10 g. of calcium fluoride, CaF_2 , with sulphuric acid, H_2SO_4 , in a lead dish. Warm this for a few seconds only. Note the irritating odors of HF which arise. Observe that they are acid to litmus paper. Place two match sticks across the top of the dish and cover it with the glass plate, wax side down. After half an hour remove the wax, and the design will be found etched in the glass.

Reaction — $\text{CaF}_2 + \text{H}_2\text{SO}_4 = \text{CaSO}_4 + 2\text{HF}$, and HF attacks the silica of the glass thus: $4\text{HF} + \text{SiO}_2 = \text{SiF}_4 + 2\text{H}_2\text{O}$.

LABORATORY QUESTIONS.

1. What per cent of KI is I?
2. What substances did you find would dissolve I?
3. Will I dissolve in KI solution? Try it.
4. Can iodine or iodides be detected by putting a drop of their solution on one's cuff?
5. Complete the equation $\text{NaBr} + \text{HgNO}_3 =$
6. Balance the equation $\text{KOH} + \text{I} = \text{KIO}_3 + \text{KI} + \text{H}_2\text{O}$.
7. What are the white fumes seen escaping in etching?
8. What becomes of the sulphur set free in experiments 48 and 51?

9. How do you explain the change of color of the CS_2 globule in experiments 48 and 51?

10. Write the reaction for preparing I from FeI_2 .

11. Why is HCl a more useful acid than HF or HI ?

12. Can you invent a method of preparing NaBr from Br and NaOH ?

13. Would Br be valuable for a disinfectant? Why?

14. How do the experiments prove Br , I and F electro-negative?

15. Arrange Cl , Br , I and F in the order of their atomic weights. Is this the order of their chemical affinity?

16. Taste the compounds of Cl , Br and I , and assign a reason why Berzelius designated them as "halogens" (salt formers).

17. How many reasons can you assign for classifying Cl , Br , I and F in one family?

18. Why cannot the hydrogen compounds of the halogens all be prepared by the action of sulphuric acid on their salts?

19. If the chlorine test be applied to a mixture of iodides and bromides, can these elements both be detected? Try it by adding small amounts of chlorine water and shaking until a large amount has been added. Note any characteristic colors in the globule.

20. Why could chlorides, bromides or iodides not be prescribed in solution with mercurous, silver or lead salts?

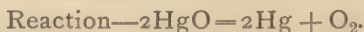
21. Compare the reactions for the preparation of Cl , Br and I from their salts. What similarity is found?

OXYGEN.

USEFUL DATA: Atomic Symbol, O ; Molecular Formula, O_2 ; Valence, 2; Atomic Weight, 16; Electro-negative; Specific Gravity, 1.1056 (Air is 1); Weight of 1 liter at 0°C and 760 m.m., 1.430 g.

54. Preparation for Mercuric Oxide. Place 1 g. of mercuric oxide, HgO , in a small test tube. Heat it very hot and

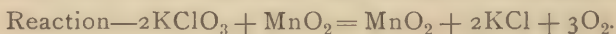
insert a glowing match stick from time to time until it bursts into flame. Examine the sides of the t.t. Notice the change of color of the remaining oxide as it cools.



55. Preparation from Potassium Chlorate. Place 2 g. of potassium chlorate, KClO_3 , in a test tube. Heat and test for O, as before. Continue to heat until the action ceases. Compare the taste of the residue with that of potassium chlorate. See 45.



56. Preparation from a Chlorate by Manganese Dioxide. Place 2 g. of KClO_3 in a test tube and mix the crystals with a little manganese dioxide, MnO_2 (fine dry sand is nearly as good). Heat and test for O, as before. Note that the gas is evolved with less heat and the MnO_2 is unchanged.



OZONE—Molecular Formula, O_3 .

57. Preparation from Ether. Put a little ether in a test tube, and keep it away from flames. Over it hold a piece of damp starch test paper¹. Heat the end of a glass rod very hot, and with it stir the ether. Ozone is formed, and turns the starch paper blue.

58. Preparation by Electricity. Notice the peculiar odor of ozone about a Holtz or other electric machine when in action. Detect its presence by holding starch test paper near the discharge.

NOTE 1. Starch test paper is made by boiling some thin starch water, dissolving in it a little KI, and in this dipping the paper. Any substance which will unite with the K will leave the I free to form blue starch iodide.

COMPOUNDS OF OXYGEN AND HYDROGEN.**Water, H_2O .**

59. Preparation by Synthesis. Prepare a hydrogen generator with an ignition jet. In it cover Zn with water and add some sulphuric acid. After it has been acting some moments, and the air is all expelled, wrap it in a cloth and light the gas. Hold over the flame an inverted beaker kept cool, if necessary, by a damp cloth wrapped about it. Notice that the moisture gathers within the beaker.



60. Composition of Water Proved by Analysis. Fill a beaker with water acidulated with sulphuric acid, H_2SO_4 . Invert in this two test tubes filled with water. Insert in the beaker the electrodes of the dynamo or battery. Collect the gases in separate tubes until a quantity of each is obtained. Note that in the same time twice as much of one gas as the other is collected. Place the thumb under the mouth of each tube in succession, invert and test the one for hydrogen by igniting the gas, and the other for oxygen by the glowing of a smouldering match.

Hydrogen Dioxide, H_2O_2 .

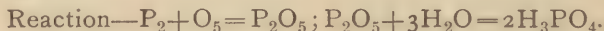
61. Preparation from Barium Dioxide. Place 10 g. of barium dioxide, BaO_2 , in a beaker of cold water. Let it stand, and stir repeatedly for half an hour. Stir, and add slowly 20 c.c. of sulphuric acid, H_2SO_4 . Let it stand, and finally filter or decant the clear solution. It contains H_2O_2 .

62. The Chromate Test for Hydrogen Dioxide. Mix in a test tube 1 drop of potassium bichromate solution,

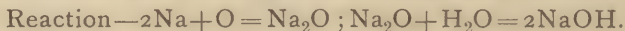
$\text{K}_2\text{Cr}_2\text{O}_7$, 2 drops of H_2SO_4 , and a little ether. Lastly, add a few drops of H_2O_2 , and shake. The supernatant ether becomes blue from the presence of perchromic acid, $\text{H}_2\text{Cr}_2\text{O}_8$, in solution.

TWO CLASSES OF OXIDES.

63. I. Acidic Oxides. Place a piece of dry phosphorus in an evaporating dish. Ignite it and quickly cover it with a glass plate, admitting as little air as is necessary to totally consume it. When action ceases examine the white powder, phosphoric oxide, P_2O_5 . It has no action on dry blue litmus paper. Add some water. The solution is acid toward litmus, and has a sour taste. Phosphoric acid, H_3PO_4 , is formed. Electro-negative chemical elements in general form oxides which unite with water, forming acids.



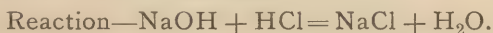
64. II. Basic Oxides. In an evaporating dish ignite a dry piece of metallic sodium. The white residue is sodium oxide, Na_2O . It has no action on dry red litmus paper. Add water. Sodium hydroxide, NaOH , is formed, which is alkaline to litmus, and has the biting taste and greasy touch of an alkali. Strong electro-positive elements form oxides, which unite with water, forming hydroxides.



Most metallic oxides are insoluble in water.

65. The Union of Acids and Alkalies. Place in a beaker a solution of sodium hydroxide, NaOH . It turns red litmus paper blue. In another beaker put some dilute HCl . It turns blue litmus red. Add one mixture to the other

until the resulting solution has no action on litmus. This is best accomplished by putting a few drops of phenolphthalein in the acid and adding the alkali drop by drop until on stirring a faint pink color appears. Evaporate this neutral solution to dryness, and taste the crystals of sodium chloride, NaCl , remaining. Acids and alkalies always unite to form a salt and water.



LABORATORY QUESTIONS.

1. How much O can be obtained from 100 g. of KClO_3 ? From 100 g. of HgO ?
2. Can you invent a method of preparing HgO ?
3. Explain the action of MnO_2 in the preparation of O.
4. Why is O "smoky" when first prepared?
5. Why does ozone turn starch test paper blue and O not?
6. What is the compound formed when iron rusts? Give the formula.
7. We can ignite Na or Mg with a match. Why not coal or iron?
8. What is meant by oxidation? What by reduction?
9. Was P or Na acid or alkaline before oxidation?
10. Why is NaOH called sodium hydroxide?
11. What is the anhydride of an acid or alkali?
12. What is formed when an acid neutralizes an alkali?
13. Can you tell whether a compound is alkaline or not by its formula?
14. Why is the amount of H collected in the electrolysis of water slightly greater than twice the O?
15. Why are O_3 and H_2O_2 chemically active substances?
16. Lime water, Ca(OH)_2 , turns green cloth yellow. How might the color be restored?
17. What should be done when acid is spilled on clothing?
18. What is an acid? What is a base? What is an alkali? What is a salt?

SULPHUR.

USEFUL DATA: Atomic Symbol, S; Molecular Formula, S₂; Valence, 2, 4 or 6; Atomic Weight, 32; Electro-negative.

FORMS OF SULPHUR.

66. Precipitated Sulphur. To half a test tube of water, add 1 g. of flowers of sulphur and $\frac{1}{2}$ g. of slaked lime. Boil and filter. Acidify the filtrate with HCl. Finely divided white sulphur is precipitated, called "milk of sulphur."

67. Crystalline Sulphur. Dissolve $\frac{1}{2}$ g. of flowers of sulphur in 5 c.c. of carbon disulphide, CS₂. Set aside until evaporated, and examine the crystals.

68. Plastic Sulphur. Melt 10 g. of roll sulphur in an evaporating dish until it is thick and dark. Pour it slowly into a bowl of cold water. Chew the plastic sulphur. It quickly turns to the crystalline variety.

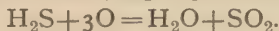
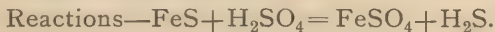
69. Test for Sulphur. Sulphur heated on a silver coin forms a black stain of silver sulphide, Ag₂S, which may be dissolved by potassium cyanide, KCy, solution.

COMPOUNDS OF SULPHUR AND HYDROGEN.

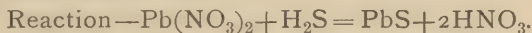
Hydrogen Sulphide, H₂S.

70. Preparation from Ferrous Sulphide. Prepare a generator with an ignition jet. In it place several pieces of ferrous sulphide, FeS. Cover them with water and add a little sulphuric acid, H₂SO₄. Note the odor of the gas evolved, and its action on moist blue litmus paper. Ignite

the jet. Note that the rotten-egg odor changes to that of burning matches. Cold porcelain, pressed down on the flame, has sulphur deposited on it.



71. Formation of Metallic Sulphides. Prepare solutions of Pb, Cu and Sb salts in separate test tubes. Pass hydrogen sulphide, H_2S , through each. Precipitates of insoluble metallic sulphides are formed.



72. Reducing Power of Hydrogen Sulphide. Pass H_2S through a test tube of strong nitric acid, HNO_3 . Sulphur is deposited. Repeat the process, using acid solutions of ferric chloride, FeCl_3 , and potassium chromate, K_2CrO_4 . The colors change to those of reduced compounds. The hydrogen unites with the O or Cl in each case.



73. Tests for Sulphides.

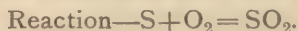
1. *The Lead Acetate Test.* Paper moistened with lead acetate solution, $\text{Pb(C}_2\text{H}_3\text{O}_2)_2$, turns black when held in H_2S , or dipped into a solution of soluble sulphides.

2. *The Sulphuric Acid Test.* Sulphuric acid, H_2SO_4 , on sulphides liberates H_2S , as in 70.

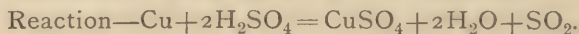
3. *The Silver Test.* A solid sulphide, mixed with sodium carbonate, fused on *porcelain*, cooled, placed on a bright silver coin and moistened with a drop of dilute HCl , yields a black stain of silver sulphide, Ag_2S , soluble in potassium cyanide, KCy , solution.

COMPOUNDS OF SULPHUR AND OXYGEN.**Sulphur Dioxide, SO₂.**

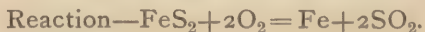
74. Preparation from Sulphur. Burn a little sulphur on the end of a match stick, and note the odor of SO₂.



75. Preparation from Sulphuric Acid by Copper. Place strong sulphuric acid, H₂SO₄, and copper turnings in a test tube. Heat until the white fumes of SO₂ arise. A moist wheat straw held in the gas will be bleached.



76. Preparation from Sulphides. Heat on an iron plate a bit of lead sulphide, PbS (galena), or iron sulphide, FeS₂ (fool's gold). Odors of SO₂ can be detected.

**Sulphurous Acid, H₂SO₃.**

77. Preparation from Water and Sulphur Dioxide. In a generator place some sodium sulphite, Na₂SO₃, and add a little hydrochloric acid. SO₂ is evolved when strong acids are added to sulphites. Pass this gas through a test tube of water. Note the odor of the solution, its taste, and action on blue litmus paper. Save the solution for experiment 82.

**78. Tests for Sulphites.**

1. *The Acid Test.* Treated with strong acids, sulphites liberate SO₂.

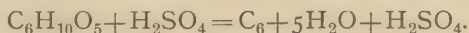
2. *The Barium Test.* BaCl₂ added to a solution of sulphites, ppts. white barium sulphite, BaSO₃. Add HCl.

BaSO_3 dissolves. Boil and filter¹. Add HNO_3 and heat. Any dissolved BaSO_3 is oxidized to insoluble barium sulphate, BaSO_4 , and precipitated.

Sulphuric Acid, H_2SO_4 .

79. Dehydrating Effect. Use dilute sulphuric acid as ink, and write on paper. When dry warm over a flame and note the charred characters. Try other acids.

Approximate Reaction—



80. Affinity for Water. In half a test tube of water pour concentrated sulphuric acid. Note the heat produced. In diluting H_2SO_4 , acid should always be added to water, not water to acid.

81. Tests for Sulphates.

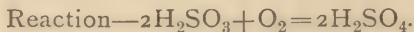
1. *The Barium Test.* BaCl_2 added to solutions of sulphates ppts. white BaSO_4 , insoluble in boiling HCl .

2. *The Silver Test.* Insoluble sulphates mixed with sodium carbonate, Na_2CO_3 , fused on *charcoal*, transferred to a bright silver coin, moistened with a drop of dilute acid, yield a black stain of Ag_2S , soluble in potassium cyanide, KCy , solution. Compare 69 and 73-3.

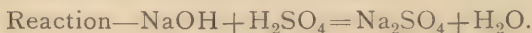
82. Illustrative Preparation of Sulphuric Acid. Take a test tube of freshly prepared H_2SO_3 , as in experiment 77. Test it for H_2SO_4 by 78-2, and set it away in an evaporating dish for some days, exposed to the air. Notice that it loses its sulphurous odor, and the test shows

NOTE 1. There will nearly always be some BaSO_4 which is insoluble, and obscures the reaction, hence it is best to boil and filter.

H_2SO_4 increasingly present. This illustrates slow oxidation.



83. Formation of Sulphates. Neutralize dilute H_2SO_4 with NaOH . Evaporate the solution to a small bulk. Cool, and examine the crystals of sodium sulphate, Na_2SO_4 , or Glauber's Salt, formed. Note how iron and copper sulphates were formed in 70 and 75.



84. Tests for Thio-Sulphates— $\text{H}_2\text{S}_2\text{O}_3$.

1. *The Hydrochloric Acid Test.* HCl , added to a solution of thio-sulphates (so called hyposulphites), ppts. S with evolution of SO_2 .

2. *The Silver Chloride Test.* Freshly prepared AgCl , 28, is dissolved by a few drops of strong sodium thio-sulphate solution, forming a very sweet syrup. Hence the use of "hypo" in photography to dissolve silver from photographic plates.

LABORATORY QUESTIONS.

1. Is sulphur soluble in water?
2. How could a solution of S be made?
3. Describe the changes observed in melting sulphur.
4. Why do eggs discolor silver spoons?
5. Why is S deposited on porcelain in example 70?
6. Volcanoes eject H_2S and SO_2 . Write the reaction showing why S is found in volcanic regions.
7. When H_2S water stands in the air S is precipitated. Write the reaction, explaining why sulphur water often contains specks of solid sulphur.
8. Is H_2S an acid? What are its salts called?
9. Complete this equation: $\text{H}_2\text{S} + \text{CuSO}_4 =$
10. Why are sulphide ores roasted in extracting the metals?

11. What is fool's gold?
 12. What are pyrites, and why so named?
 13. What per cent of SO_2 is S?
 14. Write the graphic formulas of HCl , H_2O , SO_2 and H_2SO_4 .
 15. Write the reactions for each of the tests for sulphates.
 16. Analyze a piece of rubber, and report whether sulphides or sulphates are present.
 17. How much S in 100 g. of H_2SO_4 ?
 18. What analogous compounds do S and O form?
 19. How might calcium sulphate, CaSO_4 , called gypsum, be prepared?
 20. Why should water not be poured into strong H_2SO_4 ?
 21. Why are pocket match-safes not made of silver?
 22. Which sulphides are soluble and which insoluble?
-

NITROGEN.

USEFUL DATA: Atomic Symbol, N; Molecular Formula, N_2 ; Valence, 3 or 5; Atomic Weight, 14; Electro-negative; Specific Gravity, 0.971 (Air is 1).

85. Preparation from the Air. Place a piece of phosphorus the size of a pea on a cork, which is covered with a piece of asbestos¹. Float this in a bowl of water. Ignite the P and cover it quickly with a beaker, well pressed down, so that no heated air escapes. When the action ceases, and the white fumes are all gradually dissolved in the water, cover the beaker with the hand or a glass plate, invert it and set it out, with the water it contains, on the desk. Quickly thrust in a lighted match, and note that the flame is ex-

NOTE 1. Phosphorus is kept under water. It must not be handled with the fingers, nor objects contaminated by it thrown where spontaneous combustion would be likely to cause a conflagration.

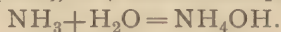
tinguished at the surface of the gas. Observe what fractional volume is occupied by water which was formerly occupied by oxygen.



COMPOUNDS OF NITROGEN AND HYDROGEN.

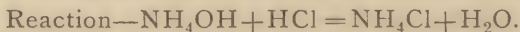
Ammonia, NH_3 .

86. Preparation from Ammonium Chloride. Gently heat in a generator a mixture of equal parts of ammonium chloride, NH_4Cl , and quicklime, CaO , taking care not to break the generator. Test the gas evolved by moist red litmus paper. Pass it for some moments through 10 c.c. of water, forming aqua ammonia.



87. Preparation from Animal Matter. In a test tube heat a few small pieces of hoof-parings from a blacksmith shop. Note the odor, and the power of the gas evolved to turn moist red litmus paper blue.

88. Formation of Ammonium Salts. Neutralize some ammonium hydroxide NH_4OH , or aqua ammonia, by adding HCl . Notice the white smoky fumes of ammonium chloride that arise. Evaporate the solution to a small bulk, and cool. Examine the crystalline ammonium chloride.



89. Tests for Free Ammonia.

1. *The Litmus Test.* Moist red litmus paper is turned blue by the fumes of NH_3 .

2. *The Ammonium Chloride Test.* A glass rod dipped in HCl and held in NH_3 yields white fumes of NH_4Cl .

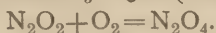
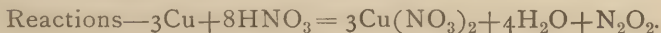
COMPOUNDS OF NITROGEN AND OXYGEN.

Nitrous Oxide, N_2O , Laughing Gas.

90. Preparation from Ammonium Nitrate. Heat some ammonium nitrate $(NH_4)NO_3$ in a generator. Nitrous oxide, N_2O , is evolved. Catch several test tubes of the gas by the displacement of water, as in 33. Note that the gas has a sweetish odor. Insert a match, and prove it a supporter of combustion.

Nitric Oxide, N_2O_2 , or NO .

91. Preparation from Nitric Acid by Copper. In a generator gently warm a mixture of nitric acid, HNO_3 , and copper turnings. Note the color of the gas evolved. Catch a test tube full of this gas by the displacement of water. Note that it is colorless. Expose it to the air, and it turns reddish brown by uniting with oxygen to form N_2O_4 , a reddish gas, one of the higher oxides of nitrogen.

Nitrous Acid, HNO_2 .

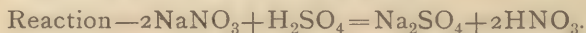
92. Preparation of Nitrites from Nitrates. Heat some dry potassium nitrate, KNO_3 , in a test tube. Oxygen is evolved, and potassium nitrite remains. Save this for the following tests. Nitrates thus readily yield oxygen.

**93. Tests for Nitrites.**

1. *The Starch Test.* Solutions of nitrites acidulated with H_2SO_4 turn blue a solution of starch-potassium-iodide mucilage (57, Note 1).

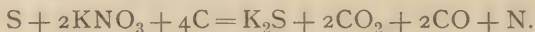
Nitric Acid, HNO_3 .

94. Preparation from Sodium Nitrate. Heat in a generator equal quantities of sodium nitrate, NaNO_3 , and H_2SO_4 . Catch 10 drops of the distillate in a test tube cooled by being wrapped in a wet cloth or immersed in a beaker of water. Put a drop of the acid on the finger nail and notice the yellow stain. Observe that it is strongly acid.



95. Preparation of Gunpowder. Take 1 part of flowers of sulphur, 2 parts of powdered carbon, and 7 parts of potassium nitrate, KNO_3 , or nitre. Dampen the mass. Grind it in a mortar and dry it. When dry, pulverize and ignite small quantities on an iron plate.

Approximate Reaction—



96. Preparation of Nitro-hydrochloric Acid. Make a mixture of 1 part HNO_3 and 3 parts of HCl in a test tube. Warm, and note the odor of chlorine liberated. Dissolve in this a scale of gold. This acid with metals forms chlorides, and its power is due to the liberation of nascent chlorine.

97. Formation of Nitrates. Heat a bit of metallic silver with HNO_3 . Dilute with water, and use the silver nitrate, AgNO_3 , in testing for chlorides, as in 43. Note how copper nitrate was formed in 91. All nitrates are soluble in water.

98. Tests for Nitrates.

1. *The Ferrous Sulphate Test.* Mix a little strong H_2SO_4 with a little fresh green ferrous sulphate solution, FeSO_4 . Cool the mixture and overlay it, in a test tube, carefully with a solution containing nitrates. Tap the tube gently, and at

the junction of the liquids a ring forms, usually brownish black, but sometimes violet, red or brown.

2. *The Indigo Test.* Indigo solution is decolorized by free nitric acid.

3. *The Nitric Oxide Test.* Any nitrate heated with H_2SO_4 and copper yields brownish red fumes of NO_2 .

LABORATORY QUESTIONS.

1. From the experiments, what per cent of air was shown to be N?
2. What is the difference between ammonia and ammonium? Aqua ammoniæ and ammonium hydroxide?
3. What per cent of NH_3 is H?
4. How might thunder storms prepare ammonia in the atmosphere?
5. Why is nitro-hydrochloric acid called "aqua regia"?
6. Why is ammonia called "spirit of hartshorn"?
7. What are spirits of ammonia?
8. Write the equation when ammonia burns in air.
9. Is quicklime an alkali? Try it on moist red litmus paper.
10. Complete the equation $\text{KOH} + (\text{NH}_4)\text{Cl} =$
11. In general, what distinctive difference is there in the action of H_2S and HNO_3 on compounds?
12. How many pounds of HNO_3 can be prepared from a ton of NaNO_3 , or Chili saltpetre?
13. Why are nitrates more easily prescribed than chlorides?
14. Complete and balance the equation $\text{N}_2\text{O} + \text{C} = \text{CO}_2$
15. How might laughing gas be manufactured from nitric acid and ammonia? Write the reactions in full.
16. Why is ammonia called the volatile alkali?
17. What difference do you observe in the color of red litmus paper which has been dipped in a fixed alkali and dried, and that of a slip dipped in volatile alkali and dried? Explain.

PHOSPHORUS.

USEFUL DATA: Atomic Symbol, P; Valence, 1, 3 or 5; Atomic Weight, 31; Electro-negative.

99. Two Forms.

(1) Waxy phosphorus should be handled with forceps. It emits a garlic odor and fumes luminous in the dark. Heat a small piece in a test tube and it burns, emitting white fumes of phosphoric pentoxide, P_2O_5 .

(2) Red or amorphous phosphorus is not luminous nor poisonous. It is an allotropic form of phosphorus. Heat a small amount cautiously in the bottom of a test tube. Some of it may then be removed on the end of a glass rod, and found to be ordinary waxy phosphorus.

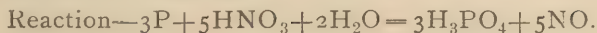
100. **Spontaneous Combustion.** Dissolve a grain of waxy phosphorus in a few drops of carbon disulphide, CS_2 . Pour the solution on a filter paper laid on a ring-stand. The paper will take fire after a few moments from the heat caused by the rapid oxidation of the phosphorus.

101. **Reducing Properties.** Add a solution of phosphorus in carbon disulphide, CS_2 , to a solution of copper sulphate, $CuSO_4$. Shake the tube, and observe the formation of black metallic copper, copper phosphate, etc., due to the union of phosphorus with the oxygen of the $CuSO_4$.

Phosphoric Acid, H_3PO_4 .

102. **Preparation from Phosphorus.** Place a small quantity of red phosphorus in an evaporating dish. Cover it with HNO_3 . Warm gently until the phosphorus is dissolved. Evapo-

rate the solution to a thick syrup. This is nearly pure phosphoric acid, H_3PO_4 .



103. Preparation from Phosphoric Oxide. Refer to experiment 63, and note the method of formation from P_2O_5 and water.

104. Formation of Phosphates. Neutralize some H_3PO_4 with sodium hydroxide, NaOH . Evaporate the solution nearly to dryness, and examine the sodium phosphate, Na_2HPO_4 , formed.

105. Precipitation of Earthy Phosphates. Dissolve bone ash in dilute HCl . Render the solution alkaline with NH_4OH , and note the precipitate of calcium phosphate, $\text{Ca}_3(\text{PO}_4)_2$.

106. Tests for Phosphates.

1. *The Silver Test.* AgNO_3 ppts. from neutral solutions of phosphates, yellow silver phosphate, Ag_3PO_4 , soluble in $(\text{NH}_4)\text{OH}$ and HNO_3 .

2. *The Magnesium Test.* A mixture, consisting of MgSO_4 , $(\text{NH}_4)\text{Cl}$ and $(\text{NH}_4)\text{OH}$ ppts. white crystalline ammonio-magnesian phosphate, NH_4MgPO_4 .

3. *The Molybdate Test.* Ammonium molybdate, $(\text{NH}_4)_2\text{MoO}_4$, ppts. from solutions of phosphates, acidulated with HNO_3 , yellow ammonium phospho-molybdate $(\text{NH}_4)_3\text{PO}_4\text{—}(\text{MoO}_3)_{10}$, which increases on application of heat and on standing.

107. Tests for Hypophosphites. H_3PO_2 .¹

NOTE 1. Phosphates are all insoluble in neutral solution, except those of the alkali bases. Hypophosphites are nearly all soluble, and are thus easily administered.

1. *The Silver Test.* AgNO_3 ppts. in neutral solutions of hypophosphites white silver hypophosphite, AgH_2PO_2 , soon turning black from the reduction of metallic silver.

2. *The Mercury Test.* HgCl_2 ppts. from solutions of hypophosphites white mercurous chloride (calomel, HgCl), when warmed with HCl .

108. **Preparation of Sodium Pyrophosphate.** Heat sodium phosphate, Na_2HPO_4 , in a test tube until the water of crystallization is given off. Gradually raise the tube to a red heat. Sodium pyrophosphate, $\text{Na}_4\text{P}_2\text{O}_7$, is formed, which will differ from the original sodium phosphate by yielding a white ppt. with AgNO_3 .



LABORATORY QUESTIONS.

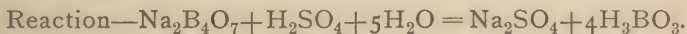
1. Is red phosphorus soluble in CS_2 ?
2. Why should oils not be given in phosphorus poisoning?
3. What is spontaneous combustion?
4. What is the anhydride of phosphoric acid?
5. Give the formulas of three acids that may be respectively neutralized by 1, 2 and 3 atoms of Na.
6. Why is H_3PO_4 said to be tribasic? How many different sodium salts can it form? Give their formulas and the reaction of each salt to litmus paper.
7. When earthy phosphates are in solution, what must be the reaction of the liquid?
8. When would urine precipitate calcium phosphate in the bladder?
9. Give the graphic formula for H_3PO_4 .
10. Why are hypophosphites useful in medicine?
11. Why is pyro-phosphoric acid so named?
12. Why are "hypo" and "ortho" phosphoric acids so named?
13. How may the latter two be distinguished by AgNO_3 ?
14. Can there be one phosphate containing three distinct bases?
15. How much P is contained in a pound of bone ash, 85% of which is $\text{Ca}_3(\text{PO}_4)_2$, and the remainder CaCO_3 ?
16. What are microcosmic salts?

BORON.

USEFUL DATA: Atomic Symbol, B; Valence, 3; Atomic Weight, 11; Electro-negative.

BORIC OR BORACIC ACID, H_3BO_3 .

109. Preparation from Borax. Make a hot saturated solution of borax, $\text{Na}_2\text{B}_4\text{O}_7$. Add sulphuric acid until slightly acid. Cool, and collect the crystals of boric acid. Note that these crystals are soluble in glycerine.



110. Borax Bead. Heat a small loop of platinum wire and dip it in powdered borax. Reheat and repeat the process until a glassy bead is obtained. Touch this with a tiny speck of cobalt compound and reheat. The bead becomes blue. Many metals may thus be distinguished by the color of the double salts formed with the borax bead.

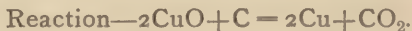
111. The Flame Test for Boric Acid. Boric acid, or borates moistened with H_2SO_4 , dissolved in alcohol and the solution ignited, tinge the flame pale green. Best seen when the flame is gently blown by the breath.

CARBON.

USEFUL DATA : Atomic Symbol, C ; Valence, 4 ; Atomic Weight, 12 ; Electro-negative.

112. Forms of Carbon. Examine the prepared samples of graphite or plumbago, lamp black, bone black, charcoal, coke, gas carbon, peat, lignite, bituminous and anthracite coal.

113. Reduction by Carbon. Mix a small quantity of powdered charcoal and copper oxide in a test tube. Heat the tube very hot and reduced, reddish metallic copper appears.



114. Decoloration by Carbon. Arrange a funnel, fitted with a filter paper and half filled with bone black. Moisten this carbon with water, and through it filter a solution of any organic coloring matter, such as litmus, indigo or cochineal. The color is removed. Repeat with a mineral coloring matter, like potassium chromate, and observe that it is not removed.

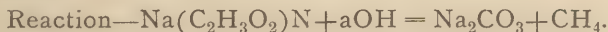
115. Deodorization by Carbon. Saturate some water with H_2S until it smells strongly of the gas. Filter this through bone black, and note that the odor is entirely removed.

CARBON AND HYDROGEN.

Methane, CH_4 .

116. Preparation from Sodium Acetate. Mix 2 g. of sodium acetate, $\text{Na}(\text{C}_2\text{H}_3\text{O}_2)$, which has been just previously thoroughly dried, with 8 g. of NaOH and 2 g. of

finely powdered quicklime, CaO . Heat this mixture very hot in a generator fitted with an ignition jet. Ignite the gas, which is methane, CH_4 , or marsh gas, the simplest of the many hydrocarbons, and the starting point in organic chemistry.



117. Preparation of Illuminating Gas. Half fill a clay pipe bowl with powdered bituminous coal. Lute the top of the bowl with wet clay or a freshly mixed plaster paris paste. Heat the bowl very hot, and at last ignite the gas at the end of the stem. Note the water given off and the coal tar produced. The vapor will turn moist red litmus blue, showing the presence of ammonia. At the end of this “destructive distillation” open the pipe and examine the coke formed.

CARBON AND OXYGEN.

Carbon Monoxide, CO .

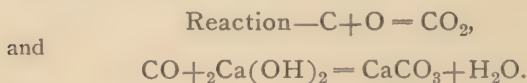
118. Preparation from Oxalic Acid. Put 5 grams of crystallized oxalic acid in a test tube and cover it with H_2SO_4 . Strongly heat the tube. After a moment the escaping carbon monoxide, CO , may be ignited at the mouth of the tube, and burns with a characteristic blue flame.



Carbon Dioxide, CO_2 .

119. Preparation by Combustion. Shake some lime water in a large test tube, and see that it remains clear. Hold a burning match in the tube until it is extinguished.

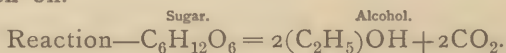
Close the tube with the thumb and shake it. Observe that the lime water becomes milky.



120. Preparation from Carbonates. In a generator cover small pieces of marble, $CaCO_3$, with dilute HCl . Collect the heavy gas flowing from the delivery tube in a beaker, as though it were water. Insert a blazing match. Observe that it is extinguished at the surface of the gas. Pour some of this carbon dioxide, CO_2 , into a tube containing lime water, shake, and confirm the presence of CO_2 .



121. Preparation by Fermentation. At the close of the laboratory period place in a generator a dilute solution of molasses and a piece of yeast. Set it in a warm place and let the delivery tube dip in a test tube of clear lime water. The next day the lime water will be turbid from CO_2 given off.

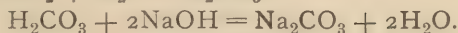


122. Detection of CO_2 in the Breath. Blow the breath through a test tube of dilute lime water. The calcium carbonate, $CaCO_3$, first formed will later redissolve. This precipitate will again fall when the solution is boiled, and the CO_2 , which holds the limestone in solution, escapes.

123. Preparation of Carbonic Acid. Pass CO_2 , from the generator prepared in the hood, through a test tube of water. The resulting solution turns blue litmus proper faintly red. Carbonic acid, H_2CO_3 , is probably formed.

When this is neutralized by NaOH, sodium carbonate, Na_2CO_3 , is produced.

Reactions— $\text{CO}_2 + \text{H}_2\text{O} = \text{H}_2\text{CO}_3$.

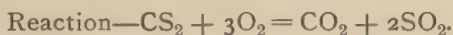


124. Test for Carbonates. Any carbonate treated with HCl effervesces and liberates CO_2 , which may be detected by passing the gas through lime water and noting any turbidity.

CARBON AND SULPHUR.

Carbon Disulphide, CS_2 .

125. Determination of Composition. Place a few drops of carbon disulphide in a bottle. Ignite it by inserting a glass rod heated to redness at the end. Note the odor of SO_2 arising, proving the presence of sulphur. When action ceases, shake the contained gas with a little lime water. Observe the turbidity as a proof of the presence of C.

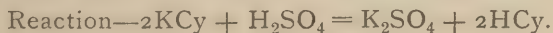


126. Solvent Power. Dissolve in CS_2 small amounts of S, rubber, oil and paraffin. Remove a grease stain from cloth with CS_2 .

CARBON AND NITROGEN.

Hydrocyanic Acid, HCN, or HCy.

127. Preparation from a Cyanide. Place a pinch of potassium cyanide, KCy, in a test tube. Cover it with dilute H_2SO_4 , warm, and note the odor of hydrocyanic acid, HCy, or Prussic acid, like the odor of peach blooms.



128. Preparation from a Ferrocyanide. In a generator place 1 g. of potassium ferrocyanide, K_4FeCy_6 , and cover

it with dilute H_2SO_4 . Gently heat the generator and pass the gas formed into a test tube of water. Dilute hydrocyanic acid, HCy , is formed. Save this for the following tests.

Reaction—



129. Tests for Cyanides.

1. *The Silver Test.* AgNO_3 ppts. from solutions of cyanides (except mercuric cyanide) white silver cyanide, AgCy , insoluble in dilute HNO_3 , soluble in NH_4OH , recognized from silver chloride by evolving the odor of HCy when treated with strong HCl (see 43).

2. *The Prussian Blue Test.* To a solution of cyanides a few drops of KOH and FeSO_4 are added, and the mixture warmed, then two drops of FeCl_3 are added. The whole is slightly acidulated with HCl , to dissolve ferrous and ferric hydroxides, when Prussian blue, $\text{Fe}_4(\text{FeCy}_6)_3$, will appear.

3. *The Sulphocyanate Test (a).* To a solution of cyanides, add 2 drops of yellow ammonium sulphide, $(\text{NH}_4)_2\text{S}_2$. Warm until colorless. Slightly acidulate with HCl , when a drop of FeCl_3 will yield a blood-red coloration. This is the most delicate test.

(b) This test may be thus performed: Place in a beaker the material containing free HCy (if a cyanide, free HCy must be liberated by adding H_2SO_4). Cover the beaker with a glass on the under side of which are two drops of $(\text{NH}_4)_2\text{S}_2$. With this the vapor forms ammonium sulphocyanate. Let the drop dry thoroughly. If HCy were present, the spot immediately turns blood-red when touched with a solution of FeCl_3 .

LABORATORY QUESTIONS.

1. How might cider vinegar be rendered white, like wine vinegar?
2. Write the reaction when iron ore, Fe_2O_3 , is mixed with coke to obtain pig iron.
3. Write the reaction showing how H_2S water was deodorized.
4. Write the reaction involved when "fire-damp" burns.
5. What is destructive distillation?
6. Write the reaction occurring when limestone is heated.
7. How is the CO_2 of the breath formed?
8. CO_2 contains O. Why does it not support combustion?
9. The yeast plant uses neither alcohol nor CO_2 . For what purpose does it split the sugar into these substances?
10. Why is NaHCO_3 called bicarbonate or acid carbonate of soda? Is it acid?
11. What other substances beside CS_2 will dissolve grease?
12. What is soda water, and how may it be prepared?
13. Name several elements that exist in allotropic forms.
14. Bone-black contains principally C, CaCO_3 , $\text{Ca}_3(\text{PO}_4)_2$, and tarry organic compounds. How may the C be obtained pure for filters?
15. If a ton of C is burned, what weight of CO_2 is produced?
16. Does wood produce as much heat during decay as during combustion?
17. Where was the C, now contained in coal, before the carboniferous age?
18. What causes beer to foam?
19. Explain the cause of a non-luminous Bunsen flame.
20. Why does burning alcohol, $(\text{C}_2\text{H}_5)\text{OH}$, not deposit soot?
21. Why do stoppers in KOH or NaOH bottles stick when left for some time? Touch the neck of one with HCl , and observe the phenomena.
22. Of what are oyster shells composed? Test one for carbonates.
23. Why is KCy used in extracting gold?
24. Why was CaO used in the preparation of methane?
25. How can a chloride be distinguished from a cyanide by AgNO_3 ?
26. Write the reaction showing how borax is a salt of tetraboric acid.
27. How may the NH_3 given off in gas manufacture be utilized?
28. What are the blue flames seen in a stove burning anthracite coal?
29. Why do kettles have crusts deposited within them?

TABLE I.

Preliminary Tests for Common Acids.

I. THE SUBSTANCE IS A SOLID.

Place a small portion in a test tube, add 1 c.c. of concentrated H_2SO_4 , and warm gently.

A. Effervescence of a colorless gas.

1. *Odorless.* Carbonates, 124; Oxalates, 213.
2. *Odor of rotten eggs.* Sulphides, 73.
3. *Odor of burning sulphur.* Sulphites, 78; Thio-sulphates, 84.
4. *Odor of peach blooms.* Cyanides, 129.
5. *Odor of vinegar.* Acetates, 209.
6. *Odor merely irritating.* Nitrates, 98; Chlorides, 43; Fluorides, 53.

B. Effervescence of a colored gas.

1. *Violet color.* Iodides, 52.
2. *Yellowish brown color.* Acrid odor, Bromides, 49.
3. *Reddish brown color.* Nitrous odor, Nitrites, 93.
4. *Greenish yellow color.* Chlorine odor, Hypo-chlorites, 166.
5. *Greenish yellow color.* Chlorine odor, with detonation, Chlorates, 45.

C. No action.

Test for H_2SO_4 , 81; H_3PO_4 , 106; HNO_3 , 98, etc.

II. THE SUBSTANCE IS A LIQUID.

Neutralize a portion, evaporate it to dryness, and test the residue by I.

TABLE II.

TABLE II.

A Table to Aid in the Detection of the More Important Acids.

A. The Substance is Soluble. Make a strong solution of a small portion. Boil it with a strong solution of Na_2CO_3 . If no ppt. occurs, no interfering bases are present. Make a solution, exactly neutralize, if necessary, and proceed by the table. If a ppt. occurs, proceed by B.

B. The Substance is Insoluble. Boil part of the powdered substance with a strong mixture of KOH and Na_2CO_3 solutions. Cool and filter. The filtrate contains all the acids present in the original substance now in the form of soluble potassium or sodium salts. *Exactly neutralize* with HNO_3 . Boil and concentrate, if dilute. Apply each of the following reagents to very small portions. Any ppts. formed will lead to the detection of the acids by the tests referred to.

Barium Chloride. Precipitates White.	Calcium Chloride. Precipitates White.	Silver Nitrate. Precipitates.	Ferric Chloride. Precipitates.	Not Precipitated.
1. <i>Insoluble in HCl.</i> Sulphates, 81.	1. <i>Soluble in much H_2O.</i> Sulphates, 81.	1. <i>Soluble in Dilute HNO_3.</i> Carbonates, reddish, 124.		<i>Test original solution.</i> Nitrites, 93.
2. <i>Effervesce with Acids.</i> Carbonates, 124. Sulphites, 78.	2. <i>Soluble in NH_4Cl.</i> Borates, 111. Carbonates, 124. Citrates, 215.	Borates, white, 111. Carbonates, white, 124. Oxalates, white, 213. Phosphates, yellow, 106.	Borates, yellowish, 111.	Nitrates, 98
3. <i>Char when Heated on Platinum.</i> Tartrates, 214. Citrates, 215.	3. <i>Insoluble in Acetic Acid.</i> Oxalates, 213. Tartrates (slightly), 214. Sulphates (slightly), 81.	Sulphites, white, 78. Tartrates, white, 214. Citrates, white, 215.	Oxalates, yellow, 213. Phosphates, yellowish, 106.	Chlorates, 45.
4. <i>Not Distinguished.</i> Oxalates, 213. Phosphates, 106. Borates, 111.	4. <i>Char when Heated on Platinum.</i> Tartrates, 214. Citrates, 215.	2. <i>Insoluble in Dilute HNO_3.</i> Chlorides, white, 43 Cyanides, white, 129 Bromides, white, 49. Iodides, yellow, 52. Sulphides, black, 73.	Sulphides, black, 73.	

THE MORE
IMPORTANT REACTIONS
ANALYTICAL AND SYNTHETICAL
OF THE PRINCIPAL
MEDICINAL METALS.

THE METALS.

THE FIVE METALLIC GROUPS.

GROUP I. THE SILVER GROUP.

Ag, Pb and Hg(ous).

130. Group Precipitant, HCl. Prepare a solution of the salts of each of these three metals in a separate test tube. Add a few drops of HCl to each, and note the white precipitates.

GROUP II. THE COPPER GROUP.

Hg(ic), Pb, Bi, Cu—As, Sb (Cd, Sn, Pt, Au).

131. Group Precipitant, H_2S in Acid Solution. Prepare a solution of the salts of each of these six metals in separate test tubes. Add a drop of HCl to each; no precipitate falls. Pass H_2S gas through each. Note the colors of the various sulphides precipitated.

GROUP III. THE IRON GROUP.

Fe, Al, Zn (Cr, Mn, Ni, Co).

132. Group Precipitant $(NH_4)_2S$ in Alkaline Solution. Prepare a solution of the salts of each of these three metals in a separate test tube. Add first a few drops of NH_4OH to each, and follow with a solution of $(NH_4)_2S$. Note the color of the precipitated Fe and Zn sulphides and Al hydrate.

GROUP IV. THE BARIUM GROUP.

Ba, Ca (Sr).

133. Group Precipitant, $(NH_4)_2CO_3$ in Alkaline Solution. Prepare a solution of the salts of each of these two metals

in a separate test tube. Add first a few drops of NH_4OH to each, and follow with $(\text{NH}_4)_2\text{CO}_3$. Note the white precipitates.

GROUP V. THE POTASSIUM GROUP.

Mg, K, Na, NH_4 (Li).

134. Group Precipitant, None. Prepare solutions of these salts, and note that they are precipitated by none of the preceding group reagents.

LABORATORY EXERCISES.

1. Test bottles Nos. 1, 2, 3, 4 and 5, and determine to which group the one salt dissolved in each belongs.

ANALYTICAL REACTIONS OF GROUP I.

135. Silver— Ag' .—*Confirm reactions, using Solution of AgNO_3 .*

1. HCl ppts. white AgCl , soluble in NH_4OH .
2. $\text{K}_2\text{Cr}_2\text{O}_7$ ppts. red Ag_2CrO_4 .
3. H_2S ppts. black Ag_2S .
4. KI ppts. pale yellow AgI .
5. NH_4OH ppts. black Ag_2O , soluble in excess.
6. NaOH ppts. brown Ag_2O , soluble in $(\text{NH}_4)\text{OH}$.
7. Solids heated with Na_2CO_3 on charcoal, in the reducing blowpipe flame, yield bright metallic beads, soluble in HNO_3 .

136. Lead, Pb'' .—*Confirm reactions, using solution of $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$.*

1. HCl ppts. white PbCl_2 , soluble in hot water and strong acids.
2. KI ppts. bright yellow PbI_2 .
3. H_2S ppts. black PbS .
4. KOH ppts. white $\text{Pb}(\text{OH})_2$, soluble in excess.

5. H_2SO_4 ppts. white PbSO_4 , in dilute solutions only on standing.

6. $\text{K}_2\text{Cr}_2\text{O}_7$ ppts. bright yellow PbCrO_4 .

7. Solids heated with Na_2CO_3 on charcoal, in the reducing blowpipe flame, yield soft metallic beads, with a yellow incrustation of PbO on the charcoal. The beads are soluble in HNO_3 .

137. Mercury (ous), Hg'' .—*Confirm reactions, using solution of HgNO_3 .*

1. HCl ppts. white HgCl (calomel), blackened by $(\text{NH}_4)\text{OH}$.

2. KI ppts. dark green HgI .

3. H_2S or $(\text{NH}_4)_2\text{S}$ ppts. black Hg_2S .

4. Bright copper, in a slightly acid solution, becomes covered with metallic Hg , made bright by rubbing.

LABORATORY QUESTIONS ON GROUP I.

1. Name the three insoluble chlorides.
2. What is a group precipitant?
3. How may silver nitrate be prepared?
4. How might one prepare lead acetate?
5. Why are silver salts used in photography?
6. Write the reaction $2\text{AgNO}_3 + 2\text{NaOH} =$
7. Why is MgSO_4 , or Epsom salt, an antidote in acute lead poisoning?
8. With what acid could Pb be dissolved from an ore?
9. Why does lead acetate added to tap water give a precipitate?
10. Try to dissolve silver stains from the hand with KCy .
11. Why is AgNO_3 an ingredient of indelible ink?
12. Make a solution containing salts of Group I. Turn to the analytical Table, III, page 77, and separate each metal.
13. Test solutions 1, 2, 3, 4, 5 and 6 on the side table, reporting the metals found in each.
14. Test ores A, B, C and D for first group metals by powdering the ore, dissolving in HNO_3 , filtering, neutralizing excess of acid, and proceeding by Table III. Report the metals found.

ANALYTICAL REACTIONS OF GROUP II.

138. Mercury(ic), Hg'' .—*Confirm reactions, using solution of $HgCl_2$.*

1. H_2S ppts. ultimately black HgS , soluble in aqua regia, insoluble in $(NH_4)_2S$.

2. $SnCl_2$ ppts. white $HgCl$, turning black with liberation of minute globules of Hg , which appear when the ppt. is boiled with HCl and rubbed.

3. $(NH_4)OH$ ppts. white $(NH_2)HgCl$ (white precipitate).

4. KOH ppts. yellow HgO .

5. KI ppts. bright red HgI_2 .

6. All mercury compounds sublime when heated, and yield reaction 4 under mercurous salts.

7. All mercury salts heated in a test tube with Na_2CO_3 give a mercury mirror.

139. Bismuth, Bi''' .—*Confirm reactions, using solution of $BiCl_3$.*

1. H_2S ppts. black Bi_2S_3 , soluble in HNO_3 , insoluble in $(NH_4)_2S$.

2. H_2O ppts. white basic $BiOCl$, when the solution with HCl , evaporated nearly to dryness, is poured into much water. The ppt. is insoluble in tartaric acid (compare Sb test, 142-4).

3. KOH or $(NH_4)OH$ ppts. white $Bi(OH)_3$, insoluble in excess, becoming yellow Bi_2O_3 on boiling.

4. K_2CrO_4 ppts. yellow $Bi_2(CrO_4)_3$, soluble in HNO_3 , insoluble in $NaOH$.

140. Copper, Cu'' .—*Confirm reactions, using solution of $CuSO_4$.*

1. H_2S ppts. black CuS , soluble in HNO_3 and KCy , insoluble in $(NH_4)_2S$.

2. NH_4OH ppts. greenish blue basic salts, soluble in excess to a dark blue solution of a double salt of copper and ammonium.

3. KOH or NaOH ppts. pale blue $\text{Cu}(\text{OH})_2$, insoluble in excess.

4. K_4FeCy_6 ppts. brown Cu_2FeCy_6 , insoluble in dilute acids, decomposed by KOH .

5. A bright steel needle dipped in a slightly acid solution of a copper salt becomes covered with metallic copper.

6. Cu salts moistened with HCl and heated in a Bunsen flame tinge it green. Solutions of Cu are always blue or green.

7. Solids fused with $\text{Na}_2\text{CO}_3 + \text{KCy}$ on charcoal, before the reducing blowpipe flame, yield bright red metallic beads, soluble in HNO_3 .

141. Arsenic, As''' .—*Confirm reactions, using solution of As_2O_3 .*

1. H_2S ppts., from acid solution, yellow As_2S_3 , soluble in $(\text{NH}_4)_2\text{S}$, KOH , NH_4OH and HNO_3 , nearly insoluble in hot HCl (compare Sb , 142-1). When As is in the form of arsenic acid, H_3AsO_4 , it must first be reduced to arsenous acid, H_3AsO_3 , by heating with HCl and Na_2SO_3 .

2. Alkalies produce no ppt.

3. AgNO_3 ppts. from neutral solutions pale yellow Ag_3AsO_3 . To a solution of As_2O_3 forming arsenous acid, H_3AsO_3 , add a few drops of AgNO_3 . Touch the surface of the solution with a glass rod moistened with dilute $(\text{NH}_4)\text{OH}$. The yellow Ag_3AsO_3 forms. With arsenic acid, the ppt. is chocolate color (151).

4. *Marsh's Test.* Fit a generator with a bent ignition jet and thistle tube, like the one on the demonstration desk. In

it generate H from pure zinc, water and H_2SO_4 . Observe the usual precaution and light the jet. The flame leaves no spot when cold porcelain is pressed in it, and no stain forms on the jet when the tube is heated to a dull redness by a Bunsen burner. Through the thistle tube add a few drops of an arsenic solution. Spots may now be deposited on porcelain, and a mirror on the tube when heated.

Tests for Arsenic Spots.

(a) They dissolve in a solution of bleaching powder.

(b) They turn yellow, touched with $(\text{NH}_4)_2\text{S}$ and evaporated. The residue is insoluble in HCl , soluble in NH_4OH .

(c) They dissolve in HNO_3 , and when this is evaporated and touched with AgNO_3 and NH_4OH and again evaporated, a brick-red spot remains (arsenic acid, 141-3).

Tests for Arsenic Mirror. Cut out a section of the tube containing the mirror, so that about 3 inches of clean tube extends beyond the stain. Hold the tube at an angle. Gently heat the mirror and drive the As into the clean portion of the tube. It will be deposited as As_2O_3 , seen under the microscope as octahedral crystals. These may be dissolved in 10 drops of hot water and tested by 141-3, or acidulated and tested by 141-1.

5. *Reinsch's Test.* Bright copper boiled with an acid solution of arsenic will be coated with metallic arsenic. Fold this up and heat it in a glass tube open at both ends. The As sublimes as As_2O_3 , which may be detected by the microscope as octahedral crystals. These may be dissolved in H_2O and tested by 141-1 and 3.

This test is a delicate and useful one, as it is unimpaired in solutions containing organic matter. Arsenates act more slowly than arsenites. Antimony and mercury

both give a deposit on copper, but Sb sublimes as an amorphous powder and Hg as minute metallic globules.

6. Solids heated in a tube with carbon, or K_4FeCy_6 , or a mixture of Na_2CO_3 and KCy, deposit metallic As on the sides of the tube.

7. Metallic As heated on charcoal burns, yielding copious white fumes of As_2O_3 , having a garlic odor.

142. Antimony, Sb'''.—*Confirm reactions, using solution of Tartar Emetic.*

1. H_2S ppts. orange Sb_2S_3 , soluble in $(NH_4)_2S$, KOH, and hot concentrated HCl (compare As, 141-1), insoluble in NH_4OH .

2. KOH ppts. Sb_2O_3 , soluble in excess.

3. NH_4OH ppts. Sb_2O_3 , insoluble in excess.

4. Solutions concentrated with addition of a few drops of HCl and poured into water ppts. white $SbOCl$, soluble in tartaric acid (compare Bi, 139-2).

5. *Marsh's Test.* Performed like test 141-4, for arsenic.

Tests for Antimony Spots.

(a) They are insoluble in a solution of bleaching powder.

(b) They turn orange when touched by $(NH_4)_2S$, and evaporated. The orange spots are insoluble in $(NH_4)OH$, but soluble in HCl.

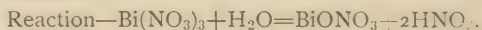
(c) They dissolve in HNO_3 , but yield no red color with $AgNO_3$ and NH_4OH (compare, 141-4, c).

Tests for the Antimony Mirror. It is often blacker than the As mirror, and yields no crystals under the microscope when treated as directed under As. The white amorphous residue is insoluble, and the attempted solution does not yield tests with $AgNO_3$ or H_2S , as under arsenic.

6. Solids fused with Na_2CO_3 on charcoal, before the reducing blowpipe flame, yield brittle metallic globules of Sb and white incrustations of Sb_2O_3 .

SOME IMPORTANT SYNTHETICAL REACTIONS OF GROUP II.

143. Preparation of Bismuth Nitrate and Sub-Nitrate. Dissolve a little powdered metallic Bi in a few drops of HNO_3 . Bismuth nitrate $\text{Bi}(\text{NO}_3)_3$ is formed. Pour this into a beaker of water. Bismuth sub-nitrate is precipitated.



144. Preparation of the Two Mercury Nitrates.

(a) *Mercurous Nitrate.* Heat a globule of Hg with 2 drops of HNO_3 . Hg is in excess, and mercurous nitrate, HgNO_3 , is formed. Dissolve this in a little distilled water, and save for 145.

(b) *Mercuric Nitrate.* Heat the Hg as above with more HNO_3 than is required to dissolve it. Mercuric nitrate, $\text{Hg}(\text{NO}_3)_2$, is formed. Dissolve in distilled water, and save for 145.

145. Preparation of the Two Mercury Iodides.

(a) *Mercurous Iodide.* To the HgNO_3 formed above, add KI. A yellowish green ppt. of yellow mercurous iodide, HgI , falls.

(b) *Mercuric Iodide.* To the $\text{Hg}(\text{NO}_3)_2$ formed above, add KI. A bright red ppt. of red mercuric iodide, HgI_2 , falls.

146. Preparation of the Two Mercury Oxides.

(a) *Mercurous Oxide.* To calomel, HgCl , add a solution of KOH, or $\text{Ca}(\text{OH})_2$. Black mercurous oxide, Hg_2O_2 , is formed.

(b) *Mercuric Oxide.* To a solution of mercuric chloride, HgCl_2 , add KOH, or $\text{Ca}(\text{OH})_2$. *Yellow mercuric oxide*, HgO , falls. Heat a few grains of $\text{Hg}(\text{NO}_3)_2$ in a test tube until fumes cease. The *red mercuric oxide*, HgO (red precipitate), remains.

147. Preparation of the Two Mercury Chlorides.

(a) *Mercurous Chloride.* Heat in a test tube equal parts of finely powdered dry NaCl and mercurous sulphate, Hg_2SO_4 . Calomel, HgCl , sublimes in the cool part of the tube. Tested by NH_4OH , it turns black.

(b) *Mercuric Chloride.* Repeat the above experiment, using mer-

curic sulphate, HgSO_4 . Mercuric chloride, HgCl_2 , or corrosive sublimate, sublimes. It is tested by dissolving some in water and adding H_2S . A black ppt. of HgS falls.

148. Test for Corrosive Sublimate in Calomel. By the above processes the products are usually mixed. Calomel is insoluble, HgCl_2 is soluble. Boil the suspected calomel, filter and pass H_2S through the filtrate. A black ppt. indicates HgCl_2 , by the formation of HgS .

149. Formation of Arsenous Acid. Note that As_2O_3 dissolves slowly in cold water, faster in hot, and also when HCl or alkalies are added.

Reaction— $\text{As}_2\text{O}_3 + 3\text{H}_2\text{O} = 2\text{H}_3\text{AsO}_3$, or arsenous acid.

150. Preparation of Fowler's Solution. Boil a grain of As_2O_3 with water containing a little K_2CO_3 , and filter if necessary. This solution of potassium arsenite, KH_2AsO_3 , colored with compound tincture of lavender and diluted to 1% of As, is Fowler's Solution. Only alkali arsenites are soluble.

151. Preparation of Arsenic Acid. Dissolve a little As_2O_3 in water, forming arsenous acid. Filter and divide into two parts. Boil one part nearly to dryness, with a little strong HNO_3 , which will oxidize arsenous acid, H_3AsO_3 , to arsenic acid, H_3AsO_4 . Arsenous acid, on addition of AgNO_3 and neutralizing with NH_4OH , yields a yellow ppt., while arsenic acid yields a chocolate ppt., 141-3. Arsenous acid is also oxidized to arsenic acid by heating thus with KClO_3 and HCl . Arsenic acid is reduced to arsenous acid by the addition of Na_2SO_3 and HCl , when SO_2 is liberated.

152. Preparation of Tartar Emetic. Precipitate a solution of SbCl_3 with Na_2CO_3 . Boil the well-washed ppt. Sb_2O_3 with an equal weight of cream of tartar in solution. Evaporate, and collect the crystals.

Reaction— $\text{Sb}_2\text{O}_3 + \text{KH}(\text{C}_4\text{H}_4\text{O}_6) = 2\text{K}(\text{SbO})(\text{C}_4\text{H}_4\text{O}_6) + \text{H}_2\text{O}$.

LABORATORY QUESTIONS ON GROUP II.

1. How many of the sulphides of the copper group are soluble in $(\text{NH}_4)_2\text{S}$?
2. Why does Pb appear in the second group?
3. Why does Hg appear in the second group?
4. What difference is there in the addition of KOH and $(\text{NH}_4)\text{OH}$ to copper solutions?

5. How does bleaching powder distinguish between As and Sb?
6. Which contains the more acid, mercurous or mercuric nitrate?
7. How might one detect pickles turned green by copper?
8. Give the formulas for arsenous and arsenic acids. Which is the more common?
9. Is there any difference in Valence in Hg(ous) and Hg(ic) compounds?
10. Write the reaction when CuO is heated with C. When As₂O₃ is heated with C.
11. $\text{H}_3\text{AsO}_3 + \text{NaOH} = ?$ Complete and balance.
12. What is an amalgam?
13. Why is calomel apt to contain corrosive sublimate?
14. Why is Reinsch's test useful?
15. What are the substances called red and white precipitates?
16. Write the reaction involved in the formation of mercuric chloride, as prepared above.
17. What substance would one get from a druggist if arsenic were called for?
18. What substance would one get from a druggist if antimony were called for?
19. Is there a difference in solubility among the varieties of white arsenic?
20. When a dime is dissolved in HNO₃, what evidence is there that it is an alloy of Cu?
21. Make a solution of CuSO₄, Pb(NO₃)₂, Bi(NO₃)₃, HgCl₂, As₂O₃ and K(SbO)(C₄H₄O₆), acidulate with HCl, precipitate the sulphides, and separate by Table III, page 77.
22. Make a solution of the first two groups, containing AgNO₃, PbNO₃ and CuSO₄. Turn to the table, and separate and identify each metal.
23. Examine bottles 7, 8, 9, 10, 11 and 12, on the side table, for metals of the first two groups, and report the metals in each.
24. Dissolve in HNO₃ the metals E, F, G and H, filter, nearly neutralize, separate by the table, and report the metals of the first two groups found.

ANALYTICAL REACTIONS OF GROUP III.

153. Iron(ous) Fe'.—*Confirm reactions, using solution of FeSO₄.*

1. NH₄OH ppts. Fe(OH)₂, white when pure, usually dirty

green from the presence of ferric salts, gradually turning, by absorption of O, to reddish brown ferric hydroxide, $\text{Fe}(\text{OH})_3$.

2. $(\text{NH}_4)_2\text{S}$ ppts. black FeS , soluble in HCl , insoluble in alkalis.

3. $(\text{NH}_4)_2\text{CO}_3$ ppts. white FeCO_3 , soluble in the presence of free CO_2 .

4. Tannic acid and tannates ppt. black iron tannate.

5. Ferrous salts are oxidized to ferric salts, slowly by standing, rapidly by heating with oxidizing agents, HNO_3 , KClO_3 , etc.

154. Iron(ic), Fe'' .—*Confirm reactions, using solution of FeCl_3 .*

1. NH_4OH or KOH ppts. reddish brown $\text{Fe}(\text{OH})_3$, insoluble in excess.

2. $(\text{NH}_4)_2\text{S}$ ppts. black FeS mixed with S, soluble in HCl and HNO_3 .

3. H_2S , in acid solutions, ppts. S and reduces "ic" to "ous" iron.

4. KCyS , K_3FeCy_6 and K_4FeCy_6 have the following reaction on "ous" and "ic" iron salts:

Reagent.	Ferrous Salts	Ferric Salts.
KCyS .	No change.	Red solution $\text{Fe}(\text{CyS})_3$.
K_3FeCy_6 .	Dark blue ppt. $\text{Fe}_3(\text{FeCy}_6)_2$.	No ppt. Reddish brown solution.
K_4FeCy_6 .	Pale blue ppt. $(\text{K}_2\text{Fe})(\text{FeCy}_6)$.	Dark blue ppt. $\text{Fe}_4(\text{FeCy}_6)_3$.

155. Aluminum, Al''' .—*Confirm reactions, using solution of $\text{Al}_2(\text{SO}_4)_3$.*

1. KOH and $(\text{NH}_4)\text{OH}$ ppt. white $\text{Al}(\text{OH})_3$, soluble in excess, but re-pptd. by the addition of ammonium salts.

2. $(\text{NH}_4)_2\text{S}$ ppts. white $\text{Al}(\text{OH})_3$.

3. Na_2HPO_4 ppts. white $\text{Al}_2(\text{PO}_4)_2$.

4. Solids fused on charcoal by the blowpipe, moistened with CoCl_2 and reheated, yield an infusible blue mass.

156. Zinc, Zn".—*Confirm reactions, using solution of ZnSO₄.*

1. (NH₄)₂S ppts. white ZnS, soluble in acids.
2. KOH ppts. white Zn(OH)₂, soluble in excess, reprecipitated on boiling.
3. (NH₄)OH ppts. white Zn(OH)₂, soluble in slight excess.
4. Na₂CO₃ ppts. white basic carbonate.
5. Solids fused with Na₂CO₃ on charcoal, in the reducing blowpipe flame, become incrustated with ZnO, yellow while hot, white when cold.
6. Solids fused on charcoal with the blowpipe, and moistened with CoCl₂ and reheated, yield an infusible green mass.

SOME IMPORTANT SYNTHETICAL REACTIONS OF GROUP III.

157. Preparation of the Two Iron Sulphates.

(a) *Ferrous Sulphate.* Dissolve iron filings or small nails in dilute H₂SO₄ until all action ceases. Filter, evaporate to small bulk and crystallize the green ferrous sulphate, FeSO₄, or iron proto sulphate.

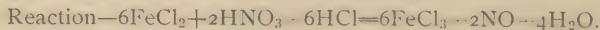
(b) *Ferric Sulphate.* Dissolve 6 parts by weight of FeSO₄ in water, and add 1 part of H₂SO₄. Heat the mixture and drop in HNO₃ until the black color first formed disappears. The resulting solution contains ferric sulphate Fe₂(SO₄)₃ or ferric tersulphate. When less acid is used the solution contains basic ferric sulphate, Fe₃O(SO₄)₅.



158. Preparation of the Two Iron Chlorides.

(a) *Ferrous Chloride.* Dissolve iron filings in HCl until action ceases. Filter, evaporate and crystallize the ferrous chloride, FeCl₂.

(b) *Ferric Chloride.* Acidulate a solution of FeCl₂ with HCl, heat and add HNO₃ until the black color first formed disappears. The resulting fluid is a solution of ferric chloride, FeCl₃.



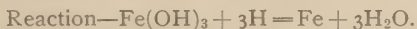
159. *Preparation of Ferrous Iodide.* Heat a few crystals of iodine with iron filings and water in a test tube. When the iodine has disappeared,

filter the greenish solution. On evaporation the solid ferrous iodide, FeCl_2 , remains.

160. Preparation of Ferric Acetate. Precipitate a solution of FeCl_3 with NH_4OH and wash the precipitated $\text{Fe}(\text{OH})_3$. Dissolve this ferric hydroxide in glacial acetic acid. The solution contains ferric acetate, $\text{Fe}(\text{C}_2\text{H}_3\text{O}_2)_3$.

161. Preparation of the Citrate of Iron and Ammonium. Prepare ferric hydroxide as above, and quickly and thoroughly wash. Dissolve it in as little of a solution of citric acid as possible, thus forming ferric citrate. Next add one-third its volume of NH_4OH . No ppt. falls, due to the formation of a double citrate of iron and ammonium. Evaporated over a water bath to a small bulk and spread on glass to dry, it forms scales, and is one of the so-called "scale" compounds of iron.

162. Preparation of Reduced Iron. Prepare a glass tube 18 inches long. In one end loosely push some CaCl_2 , in the other partly fill 4 inches of the tube with freshly prepared and dried $\text{Fe}(\text{OH})_3$. From a H generator pass the gas first over the CaCl_2 to dry it, and then over the $\text{Fe}(\text{OH})_3$. Heat the $\text{Fe}(\text{OH})_3$ by placing a Bunsen burner under the tube, and keep up the evolution of H and heat until no more moisture comes from the iron. The iron is left in the tube in a finely divided state, and when hot burns spontaneously in the air, forming Fe_2O_3 .



163. Preparation of Dialyzed Iron. Dissolve in a warm solution of FeCl_3 freshly prepared $\text{Fe}(\text{OH})_3$. Filter the solution, and place in a dialyzer and dialyze through parchment paper until the fresh diffusate gives no ppt. with AgNO_3 . Only colloid iron compounds then remain in the dialyzer, composed of about 5 per cent of basic ferric-oxy-chloride, Fe_2OCl_4 .

LABORATORY QUESTIONS ON GROUP III.

1. What is the difference in Valence in "ous" and "ic" iron?
2. What is Fe_2Cl_6 ?
3. What is blue vitriol? White vitriol? Green vitriol?
4. What action has the air on ferrous compounds?
5. How much iron in 10 grains of FeI_2 ?

6. What members of Group III are pptd. by the group reagent as sulphides? Which as hydroxides?
7. How is carbonate of iron formed?
8. Why does K_3FeCy_6 afford the best distinguishing test for "ous" and "ic" iron?
9. Why do solutions of "ous" iron usually contain "ic" iron?
10. What compound of iron is found dissolved in chalybeate waters?
11. What is the composition of black ink?
12. What is galvanized iron? Prepare by dipping bright iron wire in melted zinc.
13. How would one prepare $ZnCl_3$ for "soldering fluid"?
14. Why would sewer gas turn white paint black?
15. In what experiment did you prepare ferrous sulphide?
16. If KOH be added to excess to a solution of Fe(ic), Al and Zn, what would occur?
17. If ammonium salts were added to an alkaline solution of Al and Z, what would occur?
18. What are the blowpipe tests for Zn and Al?
19. Could dialyzed iron in the stomach enter the circulation?
20. What are the "scale" compounds of iron?
21. Write the reactions which occur when $(NH_4)_2S$ is added to solutions of third group metals.
22. Make a solution of $FeCl_3$, $Al_2(SO_4)_3$, and $ZnSO_4$; turn to Analytical Table III, page 77, and separate by methods of the third group.
23. Make a solution of $HgNO_3$, $K(SbO)C_4H_4O_6$, $Al_2(SO_4)_3$, and $ZnSO_4$, separate and identify each by Table III, page 77.
24. Test bottles 13, 14, 15, 16, 17 and 18 on the side table, and report the metals found in each.
25. Report the minerals of the first three groups found in ores I, J, K and L.

ANALYTICAL REACTIONS OF GROUP IV.

164. Barium, Ba''.—*Confirm reactions, using solution of $BaCl_2$.*

1. $(NH_4)_2CO_3$ ppts. white $BaCO_3$, soluble in acids.
2. K_2CrO_4 ppts. yellow $BaCrO_4$, insoluble in acetic acid,

soluble in HCl or HNO_3 . Ca yields no ppt. with this reagent.

3. H_2SO_4 ppts. white BaSO_4 , insoluble in hot water, acids or alkalies.

4. Ammonium oxalate, $(\text{NH}_4)_2\text{C}_2\text{O}_4$, ppts. white BaC_2O_4 , soluble in HCl and HNO_3 .

5. Solids moistened with HCl and heated in the Bunsen flame impart to it a yellowish green tinge.

165. Calcium, Ca''.—*Confirm reactions, using solution of CaCl_2 .*

1. $(\text{NH}_4)_2\text{CO}_3$ ppts. white CaCO_3 , becoming crystalline on heating.

2. H_2SO_4 ppts. white CaSO_4 from strong calcium solutions, soluble in water and acids.

3. $(\text{NH}_4)_2\text{C}_2\text{O}_4$, ammonium oxalate, ppts. white CaC_2O_4 , even in very dilute solutions, soluble in HCl and HNO_3 , insoluble in acetic acid.

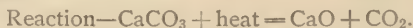
4. Solids moistened with HCl and heated in the Bunsen flame tinge it dull red, not seen in the presence of Ba.

SOME IMPORTANT SYNTHETICAL REACTIONS OF GROUP IV.

166. Preparation of Bleaching Powder. Place some damp slaked lime in a bottle and pass into the bottle Cl gas. The gas is absorbed, forming probably a loosely united compound of calcium hypochlorite and calcium chloride, thus: $2\text{Ca}(\text{OH})_2 + 4\text{Cl} = \text{Ca}(\text{ClO})_2 \cdot \text{CaCl}_2 + \text{H}_2\text{O}$. When treated with acids, the $\text{Ca}(\text{ClO})_2$ breaks up thus: $2\text{HCl} + \text{Ca}(\text{ClO})_2 = \text{CaCl}_2 + 2\text{HClO}$, and in the presence of more acid: $2\text{HClO} + 2\text{HCl} = 4\text{H}_2\text{O} + 2\text{Cl}$. When hypochlorites are treated with acids they liberate Cl, and chlorides remain in solution. Their solutions rendered acid bleach litmus solution.

167. Preparation of Quicklime. Heat a small piece of CaCO_3 or marble in the blowpipe flame, keeping it white hot for some time. Cool it, and notice that a portion of it does not effervesce with HCl.

It dissolves in H_2O , and gives the water an alkaline reaction. It is CaO , or quicklime.



168. Preparation of Plaster Paris. Heat $CaSO_4 \cdot 2H_2O$ (gypsum) in a test tube until the moisture is all given off. Considerable heat is required, but overheating must be avoided. Anhydrous $CaSO_4$, or exsiccated calcium sulphate, or plaster paris, remains, which when wet with a little water quickly hardens.

LABORATORY QUESTIONS ON GROUP IV.

1. Write the reactions occurring when lime is slaked.
2. How may calcium chloride be formed? Write the reaction.
3. Name two important uses you have thus far made of Ba compounds.
4. What is gypsum? Is it soluble in water?
5. Will moist bleaching powder give off Cl ?
6. What is the composition of slaked lime?
7. What is anhydrous gypsum called?
8. Make a solution of gum arabic (calcium gummate), and test for calcium by test 165-3.
9. What is whitening? Whitewash? Why does mortar harden when exposed to the air?
10. Will lime harden under water or protected from the air? Why?
11. How were the marbles and limestones formed?
12. How are stalactites and stalagmites formed?
13. What test best distinguishes Ba from Ca?
14. Make a solution of $BaCl_2$ and $CaCl_2$, and turn to Table III, page 77, and separate according to the scheme for Group IV.
15. Make a solution of $CuSO_4$, As_2O_3 , $FeCl_3$, $BaCl_2$, and $CaCl_2$, and separate and identify each metal by the table.
16. Test bottles 19, 20, 21 and 22 on the side table for metals of the first four groups, reporting the metals found in each.

ANALYTICAL REACTIONS OF GROUP V.

169. Magnesium, Mg'' .—*Confirm reactions, using solution of $MgCl_2$.*

1. $(NH_4)OH$ and $(NH_4)_2CO_3$ give no ppts. in presence of ammonium salts.

2. $(\text{NH}_4)_2\text{HAsO}_4$ ppts. white $\text{NH}_4\text{MgAsO}_4$ in presence of $(\text{NH}_4)\text{Cl}$ and $(\text{NH}_4)\text{OH}$.

3. Na_2HPO_4 ppts. white crystalline $\text{Mg}(\text{NH}_4)\text{PO}_4$ in presence of NH_4Cl and $(\text{NH}_4)\text{OH}$, hastened by stirring.

4. Solids fused on charcoal, moistened with CoCl_2 and reheated yield a *pink* mass. Metallic Mg burns with a brilliant white light, leaving MgO .

170. Potassium, K'.—*Confirm reactions, using a solution of KCl.*

1. PtCl_4 ppts. yellow crystalline double chloride $\text{PtCl}_4 \cdot 2\text{KCl}$ from moderately concentrated solutions, hastened by presence of alcohol.

Test thus: To 1 drop of a solution of KCl , on a microscope slide, add 1 drop of PtCl_4 and 1 drop of alcohol; stir, set aside, and examine with the microscope for yellow octahedral crystals.

2. $\text{H}_2(\text{C}_4\text{H}_4\text{O}_6)$, tartaric acid, ppts. white crystalline $\text{KH}(\text{C}_4\text{H}_4\text{O}_6)$, promoted by stirring.

3. Solids heated on platinum wire in the non-luminous Bunsen flame tinge it violet, seen through blue glass.

171. Sodium, Na'.—*Confirm reactions, using a solution of NaCl.*

1. Neither PtCl_4 or $\text{H}_2(\text{C}_4\text{H}_4\text{O}_6)$ yield ppts.

2. Solids heated on platinum wire in the non-luminous Bunsen flame tinge it intensely yellow, not seen through thick blue glass.

172. Ammonium, NH_4' .—*Confirm reactions, using solution of NH_4Cl .*

1. PtCl_4 and $\text{H}_2(\text{C}_4\text{H}_4\text{O}_6)$ ppt. as with K.

2. Heated with KOH or NaOH , fumes of NH_3 are evolved.

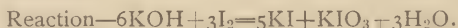
3. Nessler's reagent¹ ppts. brown NHg_2I , or in dilute solutions a yellow coloration.

4. Solids heated on platinum foil volatilize completely. K or Na salts do not.

SOME IMPORTANT SYNTHETICAL REACTIONS OF GROUP V.

173. Preparation of Potassium Hydroxide. Boil in a test tube with water equal parts of K_2CO_3 and $\text{Ca}(\text{OH})_2$, set aside, and when subsided decant the KOH solution.

174. Preparation of Potassium Iodide. Into 10 c.c. of a warm solution of KOH stir crystals of I until the solution is permanently faint yellow. Evaporate to dryness.



Heat the resulting product containing potassium iodide and iodate with an equal part of finely powdered charcoal until a slight incandescence ensues. $2\text{KIO}_3 + 3\text{C}_2 = 2\text{KI} + 6\text{CO}$. Treat the mass when cool with water, warm, filter, evaporate and crystallize out the KI.

175. Preparation of Potassium Permanganate. Mix equal parts of solid KOH, KClO_3 and MnO_2 . Fuse the mixture on platinum foil until it is dark green.



Boil this mass in water, and a purple solution of potassium permanganate results. $3\text{K}_2\text{MnO}_4 + 2\text{H}_2\text{O} = \text{K}_2\text{Mn}_2\text{O}_8 + \text{MnO}_2 + 4\text{KOH}$.

176. Preparation of Cream of Tartar. To 10 c.c. of a strong cold solution of KNO_3 add an excess of a strong solution of tartaric acid. A white ppt. falls of acid potassium tartrate, $\text{KH}(\text{C}_4\text{H}_4\text{O}_6)$, or cream of tartar. This method merely illustrates a possible method of preparation. It is not the method of commerce.

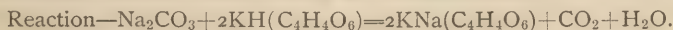
177. Preparation of Sodium Bi-Carbonate. Prepare a concentrated solution of Na_2CO_3 in less than its own weight of water. Cool this, and slowly let CO_2 bubble through the solution for some time. Set aside and

NOTE 1. To a solution of HgCl_2 add KI until the ppt. is nearly all redissolved. Render strongly alkaline with KOH. Set aside. Decant the clear straw-yellow solution for use.

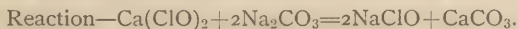
decant, drying the precipitated NaHCO_3 on filter paper. Note that an equal quantity of this acid sodium carbonate is alkaline in reaction, and effervesces more violently with acids than the original Na_2CO_3 .



178. Preparation of Rochelle Salts. To a strong hot solution of Na_2CO_3 add cream of tartar $(\text{KH})(\text{C}_4\text{H}_4\text{O}_6)$ until no further effervescence occurs. Set the solution aside to cool, and the double salt, potassium and sodium tartrate, $\text{KNa}(\text{C}_4\text{H}_4\text{O}_6)$ will crystallize out.



179. Preparation of Sodium Hypochlorite. Make a clear solution of bleaching powder. Dissolve in a little hot water twice as much Na_2CO_3 as the weight of bleaching powder used. Mix the two solutions and filter. Add water to the filtrate until the hydrometer, immersed in it, stands at 1.059. The solution (Labarraque's Solution) contains sodium hypochlorite, NaClO , a useful disinfectant, liberating Cl .



180. Preparation of Ammonium Sulphide. Pass H_2S through a solution of NH_4OH until the solution no longer gives a ppt. when a few drops are added to a little magnesium sulphate solution. Ammonium hydrosulphide is formed, NH_4HS . (Reaction $\text{NH}_4(\text{OH}) + \text{H}_2\text{S} = \text{NH}_4\text{HS} + \text{H}_2\text{O}$). To this solution add nearly an equal volume of NH_4OH . Ammonium sulphide, much used in analysis, is formed thus: $\text{NH}_4\text{HS} + (\text{NH}_4)\text{OH} = (\text{NH}_4)_2\text{S} + \text{H}_2\text{O}$. *Yellow* ammonium sulphide is usually more efficient, and is prepared by adding to ammonium sulphide a pinch of flowers of sulphur, or letting the reagent stand for some time until yellow. $(\text{NH}_4)_2\text{S}_2$ is thus formed.

LABORATORY QUESTIONS ON GROUP V.

1. What peculiar action has ammonium salts on solutions of Mg ?
2. How may both K and NH_4 be detected in a mixture?
3. What is the original source of K_2CO_3 , used in 173?
4. What is cream of tartar? Write its reaction on bicarbonate of soda.
5. How might you manufacture baking powder?
6. Why does soda sweeten sour batter cakes?
7. Why is NaHCO_3 called bicarbonate of soda?

8. What are Rochelle salts? Epsom salts? Glauber's salts?
9. In what operations has yellow $(\text{NH}_4)_2\text{S}$ or $(\text{NH}_4)_2\text{S}_2$ been used?
10. How might ammonium acetate be prepared?
11. For what purpose is NaHCO_3 more useful than Na_2CO_3 ?
12. What is pearlash? Sal soda? Saleratus? Soda?
13. What is calcined magnesia (169-4)? Is it soluble in water? Is the attempted solution alkaline? Write the reaction.
14. Make a solution of K, Na and NH_4 salts, and separate by Table III, page 77, under Group V.

TABLE III.

An Analytical Table for Separating and Identifying the More Important Medicinal Metals in Aqueous Solution.

First Group Precipitant, 1. Add HCl, drop by drop, as long as a ppt. forms; filter.

Precipitate Group I. Pb Ag Hg(ous). Wash, boil with water, filter.	Filtrate Pb. Add H_2SO_4 and set aside. White ppt. 136-5.
Precipitate Ag Hg(ous). Pour NH_4OH over the well washed ppt., on the filter.	Filtrate Pb. Add H_2SO_4 and set aside. White ppt. 136-5.
Residue Hg(ous). Black. 137-1.	Filtrate Ag. Render acid by HNO_3 . White ppt. 135-1.
Cu Add NH_4OH . Deep blue solution =	Pb Add H_2SO_4 , set aside. White ppt. =
Hg(ic) Add Cu, 137-1, or dilute and add $SnCl_2$. 138-2.	Bi Pour into much H_2O . White ppt. = Bi. 139-2.
Residue As. Yellow. Confirm by dissolving in hot HCl solution (KClO ₃). Boil off Cl and test solution by Marsh's Test. 141-4.	Filtrate Sb. H_2S gives orange ppt. or solution may be tested by Marsh's Test. 142-5.
Precipitate Fe Test the original solution by $K_2FeC_2O_8$ for "Fe" and "Pb" iron. 154-4.	Filtrate Al Zn Acidulate with HCl ; make alkaline with NH_4OH ; stir, filter.
Precip. Ba. Yellow (Con- firm by 154-5). White ppt. 155-3.	Filtrate Ca. Add $(NH_4)_2CO_3$. White ppt. 156-6.
Mg. 169-2.	White =
K Test by Flame Pt. Cl_4 . Yellow. 170-2.	Na Test by Flame Pt. Cl_4 . Yellow. 170-2.
NH_4 Test original sol. by heat with KOH . 172-2.	
Second Group Precipitant. 2 Pass H_2S through the liquid until it causes no further precipitation; warm and filter.	Cu Pb Hg(ic) Bi As Sb—Fe Al Zn—Ba Ca—Mg K Na NH_4.
Precipitate Group II. Cu Pb Hg(ic) Bi—As Sb. Wash, digest in yellow $(NH_4)_2S$, filter.	Filtrate Fe Al Zn. Third Group Precipitants. Add NH_4OH until alkaline, then $(NH_4)_2S$ and $(NH_4)Cl$, agitate, filter.
Precipitate Cu Pb Hg(ic) Bi. Wash, dissolve in aqua regia, evaporate to small bulk, cool, filter, divide into 4 parts.	Filtrate As Sb. Acidulate with $H_2C_2O_4$. Boil and filter. Ppt. = As Sb. Boil with strong HCl , dilute and filter.
Precipitate Fe Al Zn. Wash; dissolve in little aqua regia; boil to small bulk; add KOH to excess; agitate, filter.	Precipitate Ba Ca. Wash, dissolve in HCl , H_2O ; add $(NH_4)_2HAsO_4$; stir, filter.
Precipitate Ba Ca. Wash, dissolve in HCl , H_2O ; add $(NH_4)_2HAsO_4$; stir, filter.	Filtrate Ba Ca—Mg K Na NH_4 . Fourth Group Precipitant. Add $(NH_4)_2CO_3$; boil and filter.
Filtrate Mg K Na NH_4 . Add $(NH_4)_2HAsO_4$; stir, filter.	Filtrate K Na NH_4 . Evaporate; ignite; dissolve in H_2O ; divide in two parts.

EXERCISES
IN
GRAVIMETRIC AND VOLUMETRIC
PROCESSES

APPLIED TO
SANITARY WATER ANALYSIS

EXERCISES IN GRAVIMETRIC PROCESSES.

181. Determination of the Per Cent of Soluble Solids. Weigh a clean, dry evaporating dish on a good balance.¹ Fill the dish three-fourths full of the liquid under examination, as, for example, drinking water. Carefully reweigh. Evaporate the solution to dryness over a slow fire, or better, a water bath, avoiding loss by "spitting." Cool the residue by placing the dish under a desiccator.² Remove the residue, when cold, to the balance and quickly weigh. From these data compute the per cent of solids.

182. Determination of the Approximate Weight of a Precipitate. Determine the amount of lead in a solution thus: Precipitate the solution by passing H_2S through it for some time. Weigh a well-dried filter paper. Filter the solution through this paper, collecting the well-washed precipitate on it. Dry the funnel and its contents in a drying oven. Cool the dry paper and its dry precipitate in a desiccator. Weigh, and subtract the weight of the paper. The remainder is the weight of lead sulphide, PbS . From this compute the actual amount of lead in the solution.

183. Determination of the Exact Weight of a Precipitate. To determine the per cent of SO_4 in sodium sulphate, weigh accurately a small porcelain crucible. Place in it a large pinch of dry, powdered sodium sulphate. Reweigh the

NOTE 1. A good balance must be carefully handled. Special instruction in the methods of weighing, handling weights, determining centers, care of the balances, etc., will be given by the demonstrator.

NOTE 2. This is a bell-jar containing a dish of strong H_2SO_4 , which absorbs all the moisture, and keeps the material from gaining in weight.

crucible and contents. Carefully wash the salt into a beaker with about 25 c.c. of distilled water. Add a few drops of HCl, and heat to boiling. Next add BaCl_2 until no further ppt. is formed, boil and filter through a fine Swedish filter paper, which has a known ash. Wash the ppt. with distilled water until the washings no longer render AgNO_3 solution turbid, showing absence of chlorides. Dry the filter in an air bath. Fold up the dry paper compactly. Wrap a platinum wire about it. Hold it over the crucible. Burn it and shake the ash into the crucible. Heat the crucible to redness for a few minutes, until all carbon is burned and the residue is white. Cool in a desiccator. Weigh when cold. Subtract from this weight the weight of the crucible and the known filter ash. The remainder is the weight of barium sulphate. Knowing the weight of Na_2SO_4 taken and BaSO_4 found,

- (1) From the weight calculate what % of
the BaSO_4 is SO_4 _____
 - (2) From the weight calculate what % of
 Na_2SO_4 was SO_4 _____
 - Error..... _____
-

EXERCISES IN VOLUMETRIC PROCESSES.

Volumetric solutions are solutions of a definite weight of a given substance dissolved in a definite amount of distilled water. They are designated by the U. S. P. "as normal ($\frac{N}{1}$) when they contain in one litre the molecular

weight of the active reagent expressed in grams and reduced to the valency corresponding to one atom of replaceable hydrogen." A decinormal solution ($\frac{n}{10}$) is $\frac{1}{10}$ the strength of a normal one, and is preferable in many operations.

ACIDIMETRY.

184. The Process of Determining the Amount of Free Acid in a Solution.

The Solution. A decinormal volumetric solution of KOH is prepared by dissolving 5.599 grams of solid KOH in distilled water, and making up the solution to 1,000 c.c. at 15° C. In accurate work this must always be standardized.

The Process. The process is known as titration. A burette is filled with decinormal KOH solution up to the 0° mark. Place in a beaker a known quantity of the clear solution in which the free acid is to be estimated. (Use 30 c.c. of acidum hydrochloricum dilutum, and calculate the per cent of acid.) Add to the beaker 5 drops of a solution of phenolphthalein as an indicator, which turns red with the slightest excess of alkali. Place the beaker on white paper, and with constant stirring add the alkali from the burette, drop by drop, until a faint pink tinge becomes permanent. Read off the number of c.c.'s of alkali required to neutralize the acid. From the following table the exact weight of the acid may be computed. Each c.c. of alkali neutralizes exactly—

Acetic Acid, absolute $H(C_2H_3O_2)$	0.005386 g.
Citric Acid, $H_3(C_6H_5O_7)H_2O$	0.006983
Hydrobromic Acid, absolute HBr.....	0.008076
Hydrochloric Acid, absolute HCl.....	0.003637

Hydriodic Acid, absolute HI	0.012753 g.
Hypophosphorous Acid, $\text{H}(\text{H}_2\text{PO}_2)$	0.006588
Lactic Acid, absolute $\text{H}(\text{C}_3\text{H}_5\text{O}_3)$	0.008979
Nitric Acid, absolute HNO_3	0.006289
Oxalic Acid, crystallized $\text{H}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$	0.006285
Phosphoric Acid, absolute H_3PO_4	0.004890
Sulphuric Acid, absolute H_2SO_4	0.004891
Tartaric Acid, crystallized $\text{H}_2(\text{C}_4\text{H}_4\text{O}_6)$	0.007482

ALKALIMETRY.

185. The Process of Determining the Amount of Free Alkali in a Solution.

The Solution. A decinormal volumetric solution of oxalic acid, $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$, is prepared by dissolving 6.285 grams of pure crystals in distilled water, and making up to 1,000 c.c. at 15°C . In accurate work this solution must always be standardized.

The Process. Fill a burette with decinormal oxalic acid solution up to the 0° mark. Place in a beaker a known amount of any clear solution in which the amount of free alkali is to be determined. (Use 100 c.c. of liquor calcis, and prove that it contains 14% of $\text{Ca}(\text{OH}_2)$, as required.) Color the solution yellow by a few drops of methyl-orange solution for an indicator, which turns red with the slightest excess of acid. With constant stirring, drop in the oxalic acid from the burette until the pink color appears. Read off the number of c.c.'s of acid used, and compute the absolute weight of alkali. Each c.c. of oxalic acid neutralizes exactly—

Ammonia Gas, NH_3	0.001701 g.
Ammonium Carbonate, $(\text{NH}_4)_2\text{CO}_3$	0.004793

Calcium Hydroxide, $\text{Ca}(\text{OH})_2$	0.003691 g.
Potassium Hydroxide, KOH	0.005599
Sodium Bicarbonate, NaHCO_3	0.008385
Sodium Carbonate, Na_2CO_3	0.005292
Sodium Hydroxide, NaOH	0.003996

186. Estimation of Chlorine.

The Solution. A standard volumetric solution of silver nitrate convenient in water analysis is prepared by dissolving 4.79 grams of pure AgNO_3 in distilled water and making it up to 1,000 c.c. at 15°C . This solution is of such strength that 1 c.c. of it precipitates exactly one milligram of chlorine (or 1 c.c. is equivalent to .00165 g. of NaCl). The solution must be kept in a dark container.

The Process. Fill a clean burette to the 0° mark with the standard solution of silver nitrate. Place a beaker on a piece of white paper. In it put a known quantity of the clear solution in which chlorides are to be determined. (Take 70 c.c. of drinking water.¹) Add a crystal of K_2CrO_4 to color the water lemon-yellow. With constant stirring, drop in the silver nitrate from the burette until a faint red color is permanent. Read the number of c.c.'s used. Refill the burette and repeat the process with an equal volume of distilled water exactly matching the red color. Read the number of c.c.'s used, and deduct this from the first reading, as the amount required to produce the color. The number of c.c.'s remaining represents grains of chlorine per gallon, or may be calculated as sodium chloride.

NOTE 1. Seventy c.c. of distilled water weighs 70,000 milligrams. The imperial gallon contains 70,000 grams, hence the number of milligrams of solids in 70 c.c. will likewise express the number of grains in a gallon.

SANITARY

ANALYSIS OF POTABLE WATERS.

187. Detection of Poisonous Minerals. Poisonous metals most commonly present in potable waters are lead and copper, sometimes arsenic, zinc, etc. A quantity of water is evaporated to a small bulk. The metals may be then identified by the general methods of qualitative analysis given in Table III, page 77.

188. Determination of Total Solids. Find the total solids in 70 c.c. of water, according to the method in 181. The number of milligrams of solids likewise expresses grains per imperial gallon. River waters usually contain less solids than well waters. Solids ought not to run over 30 or 40 grains per gallon (Wanklyn). Artesian water often runs higher. The presence of carbonates in the residue is evinced by effervescence when touched with acid.

189. Determination of Chlorine. Find the number of grains of chlorine per gallon, as in 186. Pure waters, unless charged from some uncommon mineral deposits, contain little chlorine. Urine and sewage contaminated from animal excretions are highly charged with chlorides. A water free from chlorides could not have been contaminated. Water containing much chlorine would be looked upon with suspicion until its albuminoid ammonia had been determined. River water contains not far from 1% of chlorides. Well water contains more. Five or six grains of chlorine per gallon does not injure water, but is a reason for suspicion.

190. Determination of Free and Albuminoid Ammonia.

I. *Distillation.* Thoroughly cleanse a Liebig condenser and a glass retort holding 1 litre. Arrange them for distillation. In the retort place 500 c.c. of the water under examination. Add a handful of broken, well-washed glass, to keep from "bumping." Slowly distill, catching each 50 c.c. of distillate in a large tube, and set each aside in the order of distillation until four tubes are filled, representing 200 c.c. These contain any free ammonia in the original portion, and are to be examined later by (a).

Stop the distillation. Add 50 c.c. of permanganate solution¹ through a funnel, and begin to distill very cautiously to avoid bumping. Catch each 50 c.c. of distillate in a large tube, and set each aside as before until three are obtained. These contain ammonia from any organic matter present in the original solution, and are to be examined later for albuminoid ammonia by (b).

II. *Nesslerizing.* Prepare two solutions of ammonium chloride.

1. 3.15 g. of NH_4Cl dissolved in 1 litre of distilled water. 1 c.c. contains 1 milligram of ammonia.

2. Dilute 1 c.c. of the above with 99 c.c. of water. 1 c.c. contains $\frac{1}{100}$ of 1 milligram of ammonia.

(a) *Estimation of Free Ammonia.* Take the tubes containing the free ammonia distillate. Into the first drop 2 c.c. of Nessler's reagent (172-3, Note 1), and stir. It will strike a yellow or brownish color if traces of ammonia are present, assuming its deepest permanent hue in 3-5 minutes.

The next process consists in making an artificial solution,

NOTE 1. The permanganate solution is prepared by dissolving 200 grams of solid potassium hydroxide and 8 grams of crystallized potassium permanganate in water, boiling for 20 minutes and making up to 1,000 c.c. with distilled water. This solution oxidizes organic matter and, in the presence of an alkali, liberates N in the form of NH_3 .

with a known amount of ammonia, that strikes the same color with Nessler's reagent. Take a tube containing about 45 c.c. of water free from ammonia. Add for the first trial, say 3 c.c. of the dilute ammonium chloride solution. Stir. Add 2 c.c. of Nessler's reagent. Stir, and compare the two colors by looking down through the solutions on a white surface. If the colors do not exactly agree in depth of shade, make another solution with more or less ammonium chloride, which will exactly match the distillate. The amount of ammonia added, obviously, is the amount contained in the first 50 c.c. of distillate.

The remaining tubes might be Nesslerized, but as the first tube invariably contains three-fourths of the entire free ammonia, it is easier to add one-third for the total amount of free ammonia contained in the 500 c.c. of water examined.

(b) *Estimation of Albuminoid Ammonia.* Nesslerize, in the manner described, each tube containing albuminoid ammonia, and add the results for total albuminoid ammonia in the 500 c.c. of water taken.

(c) *Discussion of Results.*

EXAMPLE OF ANALYSIS.

IN 500 C.C.		
Free ammonia.....	.01	milligram.
Correction, $\frac{1}{3}$003	"
Total013	"
Albuminoid ammonia.....	.035	milligram.
" "015	"
" "000	"
Total050	"
IN ONE LITRE.		
Free ammonia.....	.026	milligram.
Albuminoid ammonia10	"

If water contains 0.00 parts of albuminoid ammonia per million (that is, milligrams per litre), it is organically pure. If it contains 0.02–0.05 it is classed with good waters. 0.10 begins to be a suspicious sign, and 0.15 ought to condemn the water (Wanklyn). In river waters, where the organic matter is from vegetable origin, even a somewhat higher amount of albuminoid ammonia might exist, however, without serious effects.

191. Determination of Other Ingredients. Many other determinations are sometimes made in sanitary analysis of water, which may serve for valuable deductions concerning water supply, etc., such as the determination of nitrites, nitrates, hardness, bases, acids, oxygen-consuming power, etc. The above, however, are all that are usually required in determining the purity of water. The residue from concentration may be used in determining the bases and acids present by the ordinary processes of analysis. See Tables I, II and III. For further work, the student is directed to Wanklyn's **Water Analysis**.

LABORATORY EXERCISES
IN THE
MEDICINAL
ORGANIC COMPOUNDS.

ORGANIC CHEMISTRY.

192. Determination of the Fusing Point. Select a fine capillary tube, of an internal diameter just sufficient to insert a fine wire. Seal one end. Push a very small amount of the substance under examination into the lower end of the tube. Fasten the tube to a good chemical thermometer by a rubber band, and insert the whole in a beaker of water. Apply a gentle heat to the beaker, and as the temperature rises carefully note the reading of the thermometer the moment the solid melts. Whenever the fusing point is above 100°C. , a beaker of melted paraffin may be used. Any impurity will account for variations in the fusing points. Determine the fusing points of the following substances :

Substance.	Observed.	Given.
Chloral hydrate		57°C.
Naphthalene		79°C.
Stearic acid		69°C.

193. Determination of the Boiling Point. Place about 10 c.c.'s of the liquid under examination in a dry, clean 7-inch test tube, fitted with a 2-hole rubber stopper, holding a thermometer, which should nearly touch the surface of the liquid, and be free from the sides of the tube. Place a few bits of broken glass in the liquid to assist in the boiling. Place the whole in a beaker of water or melted paraffin, as required. Apply a gentle heat, and carefully note the reading of the

thermometer when the liquid is boiling vigorously and the thermometer stem is thoroughly surrounded by vapor. Determine the boiling point of the following substances. When the determination is made, return the liquid to its container.

Substance.	Observed.	Given.
Ethyl Alcohol		78° C.
Methyl Alcohol		66° C.
Ether		35° C.

194. Optical Activity of Organic Chemicals. Examine with the polariscope the prepared solutions of cane sugar, which is dextro-rotary, and morphine hydrosulphate, which is laevo-rotary.

195. Detection of Carbon and Hydrogen in Organic Compounds. Dry and powder the substance under examination, as, for example, tartaric acid. Mix it with twice its bulk of dry, finely pulverized cupric oxide, CuO . Place the mixture in a test tube, fitted with a delivery tube, constructed so that any gas formed may pass through a glass tube, in which is loosely placed a few pieces of dry CaCl_2 , and then bubble through clear lime water. Heat the test tube to a dull red. The moistening of the CaCl_2 indicates H_2O , and a precipitate in the lime water, CO_2 , both formed from the union of the H and C of the tartaric acid with the O of the CuO . Note that metallic copper remains in the test tube.

196. Detection of Nitrogen in Organic Compounds. Place in a dry test tube a small piece of metallic sodium. Cover this with the dry powdered solid under examination, as, for example, urea or any alkaloid, and heat. Cyanides are

formed if the compound contains nitrogen. Test the residue for cyanides by adding water, filtering and boiling with a drop each of FeSO_4 and NaOH , then adding a drop of FeCl_3 and acidulating with HCl . Cyanides yield a ppt. of Prussian blue, which is best seen on the filter paper when the solution is filtered (129).

197. Detection of Sulphur in Organic Compounds.

(1) Take some lead acetate solution in a test tube. Add KOH until the precipitate first formed dissolves. In this drop a little finely powdered organic substance containing sulphur, like albumin. Boil, and notice the blackening of the sulphur compound. A still more delicate test is the following:

(2) In a dry test tube place a small piece of metallic sodium. Cover this with the dry powdered substance under examination, like albumin, and heat. If the compound contains sulphur, sodium sulphide, Na_2S , will be formed. After prolonged ignition, dissolve this residue in water. Filter, and add a few drops of sodium nitro-prusside, which yields with sulphides a purple coloration.

198. The Determination of Formulæ. Examine the combustion furnace with the combustion tube, containing an intimate mixture of CuO and .46 g. of acetic acid. Note the arrangement of the weighed CaCl_2 tubes to retain the H_2O . Evidently $\frac{1}{9}$ their increase in weight will be the weight of H in the compound. Note the weighed potash bulbs arranged to absorb all CO_2 . Evidently $\frac{3}{11}$ of their gain in weight will represent the C in the acetic acid. The remaining weight of the substance we know to be O , and its amount can be determined by the method of difference.

Result of the Combustion of .46 g. of Acetic Acid.

$$\text{CO}_2 = .6765 \text{ g.} \times \frac{3}{11} = .1845 \text{ g. of C, or } 40.11\%$$

$$\text{H}_2\text{O} = .2817 \text{ g.} \times \frac{1}{9} = .0313 \text{ g. of H, or } 6.82\%$$

$$\text{O by difference } 53.07\%$$

$$\underline{\underline{100.00}}$$

This does not give us the formula. The weight of one molecule is determined by the Victor Meyer vapor density apparatus. Examine this instrument, with its outer casing in which aniline has been boiled, and the inner tube surrounded by hot aniline vapor. Note the little bottle in the inner tube, which was dropped in when the apparatus was hot. It contained .09 g. of acetic acid, which vaporized and crowded out 33.48 c.c. of air into the graduated tube. 33.48 c.c. of acetic acid vapour, then, weighs .09 gram. 33.48 c.c. of H weighs .00299 gram (1 c.c. = .0000896 g.). Hence the acetic acid vapour is about thirty times as heavy as an equal volume of H ($\frac{.09}{.00299} = 30$). Avogadro's law states that equal volumes of gases under equal conditions contain equal numbers of molecules. Then a molecule of acetic acid weighs thirty times as much as a molecule of hydrogen (H_2), and 60 times an atom of hydrogen (H). The molecular weight of acetic acid, then, is 60, and the formula is thus calculated:

Computation of Formula, Given Percentage Composition and Molecular Weight.

$$\text{C} = 40.11\% \times 60 = 24.06 \div 12 = 2$$

$$\text{H} = 6.82\% \times 60 = 4.09 \div 1 = 4$$

$$\text{O} = 53.07\% \times 60 = 31.84 \div 16 = 2$$

Thus the formula of acetic acid is $\text{C}_2\text{H}_4\text{O}_2 = 60$.

LABORATORY QUESTIONS.

1. Of what value is the estimation of the boiling and fusing points?
2. In the determination of boiling points, why should the thermometer not touch the liquid?
3. What are the causes of inaccuracy in determinations of boiling and fusing points?
4. Of what value is a knowledge of the optical activity of substances?
5. Why is the detection of N often valuable?
6. Deduce the formula for alcohol with a molecular weight of 46, when C = 52.18% ; H = 13.04% ; O = 34.78%.
7. Sulphuric acid has a molecular weight of 98. Deduce the formula when H = 2.04% ; S = 36.65% ; O = 65.31%.
8. Chloroform has a composition C = 10.04% ; H = .84% ; Cl = 89.12%. What are some possible formulas? How would its formula be decided?
9. What are some of the difficulties which keep compounds from having their formulas accurately determined?

199. Determination of the Flashing Point of Kerosene. Fill a 7-inch test tube one-third full of kerosene. Insert a thermometer, also a bent glass tube so arranged that air can be blown through the oil and bubbles of foam kept constantly upon its surface. Place the test tube in a beaker of water. Apply a gentle heat, so that the temperature rises 1° in two or three minutes. At each rise of 1° , blow for a few seconds through the glass tube until foam stands on the oil, then apply a flame to the mouth of the tube. When the flame flashes down to the oil, the reading of the thermometer gives the flashing point. A second determination, with a fresh sample, will enable a more accurate result to be secured. The laws should require oil to have a flashing point above the maximum temperature of the atmosphere in the locality where it is used.

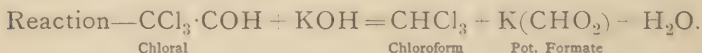
200. Preparation of Iodoform. In half a test tube of water put 2 c.c. of alcohol and 3 c.c. of KOH. Warm and add

crystals of metallic iodine, with constant stirring, until a fine yellow powder becomes visible. Filter the solution, and examine the crystals of iodoform, CHI_3 , on the filter paper.

Reaction—



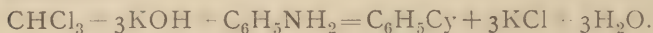
201-a. Preparation of Chloroform. In a generator put about 3 grams of chloral hydrate, cover with KOH solution, and warm and condense the vapor in a test tube immersed in a beaker of ice water. Some water will be condensed with a globule of pure chloroform, CHCl_3 , which will have the chloroform odor, and, when poured on the hand, will quickly evaporate. The purest chloroform is prepared by this method. It may also be prepared by the action of alcohol and bleaching powder.



201-b. Tests for Chloroform.

1. *The Iso-nitrile Test.* In a test tube take a little alcohol and KOH. Add a drop of chloroform and a drop of aniline. Warm, and the peculiar disagreeable, persistent odor of benzyl-iso-cyanide is developed.

Reaction—



2. *The Flame Test.* Prepare and light a H generator. A rod, wet with ammonia, above the flame gives no fumes, and a copper wire in the flame does not color it, except momentarily. Through the thistle tube add a drop of chloroform. The Cl escapes as HCl , and yields fumes of ammonium chloride, with the rod wet with ammonia, and the wire heated colors the flame green from the constant formation of CuCl_2 .

202. Preparation of Alcohol by Fermentation. Alcohol may be prepared from the fermentation of molasses or sugar, but the following process illustrates the usual commercial method. In a saucepan, or large evaporating dish, boil a quart of water. Into this slowly sift a mixture of flour and cornmeal, with constant stirring, until a thin, even mush is produced. Cool this to 60°C , and put into a large flask or bottle. Next pound up some dry malt (see 254). Add water and filter out the husks. Add the solution to the mush. Stir, and notice how rapidly the mush liquefies as the diastase of the malt converts starch to malt sugar. Next add some yeast to the solution. Close the bottle with a perforated cork, in which a delivery tube is so fitted that it dips into a beaker of lime water. Notice that CO_2 is soon evolved, as indicated by the turbidity of the lime water. When the action nearly ceases, after about two days, examine a few drops of the solution under the microscope, and note the form of the yeast plant (*Saccharomyces cerevisiae*). Next filter the solution into the distillation flask, and distil in the apparatus prepared by the demonstrator. Note the temperature at which distillation begins, and continue until the thermometer rises to about 96° . Test this distillate by the iodoform test, 204.

This distillate can be shaken with quick lime, which removes water, unites with any acetic acid formed, and decomposes any etherial salts. It can then be filtered and redistilled. The strongest alcohol obtained by mere distillation is called "rectified spirits," and contains about 7% of water.

203. Absolute Alcohol. Heat a few grams of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) until it loses its water of crystallization, and is white. Shake the powder with alcohol. After

standing, if water is present, the blue color is restored to the salt. This test for water in alcohol affords a convenient method of preparing nearly absolute alcohol, but it contains some CuSO_4 in solution, which, however, does not interfere with its use in preserving and hardening specimens, etc. It may be purified by distillation. Commercially, absolute alcohol is prepared by distilling alcohol from quicklime, adding a little metallic sodium and redistilling.

204. The Iodoform Test for Alcohol. Warm a portion of the suspected alcohol with KOH . Add crystals of I , and stir as long as the iodine is decolorized, or until a yellow powder appears. Cool and set aside. If alcohol were present, yellow crystals of iodoform, CHI_3 , precipitate, and can be seen by the microscope as hexagonal stars and rosettes. Some other organic compounds, like acetone, aldehyde, etc., yield the iodoform test, but do not cause confusion in testing potable liquids.

205. Detection of Alcohol in Beer, Etc. Distill a small quantity of beer, or any alcoholic solution under examination. Test the first portion of the distillate for alcohol by the iodoform test, 204. Beer contains from 1-4% alcohol. The quantity of alcohol is usually determined from the specific gravity of a measured quantity of the distillate.

LABORATORY QUESTIONS.

1. What would be a safe flashing point for Texas oil?
2. Why is a lamp in which oil is low more liable to explode than when filled?
3. Is kerosene explosive? Insert a lighted match into a beaker of oil, and see.
4. Give the chemical names of chloroform and iodoform?
5. What is a substitution product?
6. What relation does alcohol bear to an alkali?

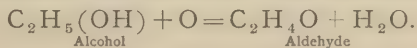
7. For what purposes is absolute alcohol useful?
8. When alcohol is in contact with specimens, what change does it undergo? How would you preserve a specimen in alcohol?
9. What is the cause of the blue color of CuSO_4 ?
10. Write the reaction showing how lime assists in the preparation of absolute alcohol?
11. How does the preparation of whisky differ from that of alcohol?
12. What are alcoholic "tears," observed in the distillation of alcohol, and why formed?

206. Preparation of Ether. Into a generator put 20 c.c. of alcohol. Keep it cool, and slowly add 10 c.c. of strong H_2SO_4 . Mix, gently warm, and catch the distillate in a test tube surrounded by a beaker of ice and water. The distillate will be ether, $(\text{C}_2\text{H}_5)_2\text{O}$, mixed with a little alcohol. Pour the distillate on the hand. Note the odor and the cold produced.



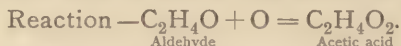
207. Preparation of Aldehyde. In a generator mix 20 c.c. of a solution of potassium bichromate, $\text{K}_2\text{Cr}_2\text{O}_7$ and 2 c.c. of H_2SO_4 . Cool the mixture. Next add 5 c.c. of alcohol, heat, and catch several c.c.'s of the distillate in a test tube, cooled by being immersed in a beaker of ice and water. The distillate has the sharp odor of ethyl aldehyde, $\text{C}_2\text{H}_4\text{O}$, and is mixed with some alcohol.

Reactions— $\text{K}_2\text{Cr}_2\text{O}_7$ and H_2SO_4 liberate O.



Test a few drops by 209-2 for acetic acid. There is none present. Set the remainder away, exposed to the air, for some

time. Aldehyde is slowly oxidized to acetic acid. Test the solution for acetic acid by 209-2, and prove its presence.



This process illustrates the oxidation of alcohol to acetic acid, which is performed in one stage by the mother of vinegar (*bacterium aceti*).

208. Tests for Aldehydes.

1. *The Silver Test.* Take a solution of AgNO_3 in a test tube. Add 3 drops of NH_4OH and a few drops of an aldehyde, as, for example, formaldehyde, CH_2O , called formalin, a powerful non-poisonous preservative and antiseptic. Next warm the solution and set it aside. The aldehyde will reduce the silver nitrate and deposit a bright mirror of metallic silver on the sides of the tube.

2. *The Potassium Test.* To a solution of KOH add a few drops of an aldehyde and heat gently. A yellow aldehyde resin, having a peculiar odor, is precipitated.

209. Tests for Acetates.

1. *The Sulphuric Acid Test.* Heated with H_2SO_4 , acetates evolve fumes of acetic acid, $\text{H}(\text{C}_2\text{H}_3\text{O}_2)$.

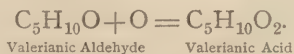
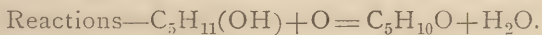
2. *The Ferric Chloride Test.* FeCl_3 forms in exactly neutral solutions a deep red liquid, due to the presence of red $\text{Fe}(\text{C}_2\text{H}_3\text{O}_2)_3$, which when boiled precipitates red oxacetate of iron, $\text{FeO}(\text{C}_2\text{H}_3\text{O}_2)$.

3. *The Acetic Ether Test.* Strong solutions of acetates mixed with H_2SO_4 and a little alcohol, when heated evolve the peculiarly fragrant odor of ethyl acetate, or acetic ether, $\text{C}_2\text{H}_5(\text{C}_2\text{H}_3\text{O}_2)$.

210. *The Detection of Mineral Acids in Vinegar.* Take a dilute solution of methyl violet. Add a drop of acetic acid or

other organic acid. No change occurs. Now add a few drops of acetic acid containing a minute amount of H_2SO_4 , or any mineral acid. The color immediately changes from violet to blue, and to green when the mineral acid is in excess.

211. Preparation of Valerianic Acid. Into a generator put 20 c.c. of $\text{K}_2\text{Cr}_2\text{O}_7$ and 2 c.c. of H_2SO_4 . Cool the mixture. Next add 7 c.c. of amyl alcohol. Heat, and catch the distillate in a cool tube. The substance is composed of a mixture of valerianic aldehyde, $\text{C}_5\text{H}_{10}\text{O}$, valeric acid and water. Cool the generator, pour back the distillate, and distil again. The second distillate is purer valerianic acid, $\text{H}(\text{C}_5\text{H}_9\text{O}_2)$, with its characteristic odor and acid reaction.



212. Preparation of Oxalic Acid. Heat a mixture of 10 parts of strong HNO_3 and 2 parts of cane sugar until nitrous fumes cease to be evolved. On cooling, crystals of oxalic acid, $\text{H}_2\text{C}_2\text{O}_4$, are deposited. On a large scale, oxalic acid is prepared by heating sawdust and soda.

213. Tests for Oxalates.

1. *The Barium Test.* BaCl_2 slowly ppts. from neutral solutions a white ppt. of BaC_2O_4 , slightly soluble in acetic acid, soluble in HNO_3 , HCl and NH_4Cl .

2. *The Calcium Test.* CaCl_2 slowly ppts. white CaC_2O_4 , insoluble in acetic acid, soluble in HCl and HNO_3 .

3. *The Silver Test.* AgNO_3 ppts. white $\text{Ag}_2\text{C}_2\text{O}_4$, soluble in HNO_3 and NH_4OH .

4. *The Carbon Monoxide Test.* Heated in a test tube with H_2SO_4 ,

oxalates without charring evolve CO, which, when ignited, burns at the mouth of the tube with its characteristic blue flame.

Heated alone in a test tube, oxalates without charring evolve CO, and the white residue effervesces on addition of HCl.

214. Tests for Tartrates.

1. *The Calcium Test.* CaCl_2 , after a few moments, ppts. white crystalline $\text{Ca}(\text{C}_4\text{H}_4\text{O}_6)$. NH_4Cl prevents this precipitation. The ppt. is soluble in KOH, but reprecipitated on boiling. It is slightly soluble in acetic acid.

2. *The Barium Test.* BaCl_2 ppts. white $\text{Ba}(\text{C}_4\text{H}_4\text{O}_6)$, soluble in HCl and NH_4Cl .

3. *The Silver Test.* AgNO_3 ppts. in neutral solution white $\text{Ag}_2(\text{C}_4\text{H}_4\text{O}_6)$. This ppt. blackens on boiling, and is soluble in HCl and NH_4Cl .

4. *The Heat Test.* Tartrates char when heated alone or with H_2SO_4 .

215. Tests for Citrates.

1. *The Calcium Test.* CaCl_2 in neutral solution precipitates perfectly, on boiling, $\text{Ca}_3(\text{C}_6\text{H}_5\text{O}_7)_2$. Unlike calcium tartrate, this calcium citrate is insoluble in KOH.

2. *The Silver Test.* AgNO_3 ppts. white $\text{Ag}_3\text{C}_6\text{H}_5\text{O}_7$, and, unlike silver tartrate, this ppt. does not blacken on boiling.

3. *To detect a Mixture of Tartrates and Citrates.* Precipitate the mixture with CaCl_2 , heat, filter and digest the well-washed ppt. with cold KOH. Dilute and filter. The filtrate contains the tartrate, which is precipitated on boiling. The undissolved residue on the filter contains the citrate. Dissolve this in NH_4Cl solution, filter, and the calcium citrate will precipitate when boiled.

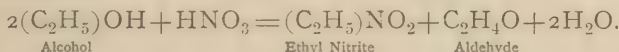
LABORATORY QUESTIONS.

1. How would you detect aldehyde in alcohol?
2. Can you mention an insoluble normal acetate?
3. Is acetic ether a proper name for ethyl acetate?
4. What is an ester?
5. What is an ether? What is an alcohol?

6. What is an aldehyde?
7. How may you distinguish between HCl and $\text{H}_2\text{C}_2\text{O}_4$ by AgNO_3 ?
8. What acid does a Seidlitz powder contain? Test one.
9. What substance is the best for use to preserve pathological specimens?
10. Show the steps in the change from alcohol to vinegar.
11. Of what value is brown paper or tea leaves added to dilute molasses in vinegar making?
12. How does oxalic acid aid in removing inkstains?
13. Why does wine sour when the bottle is not well corked?
14. Is oxalic acid in pieplant poisonous? Why?
15. Is there any reason why vinegar should contain a mineral acid?
16. What relation does chloral bear to aldehyde? Will it give the mirror test for aldehydes, 208-1? Try it.

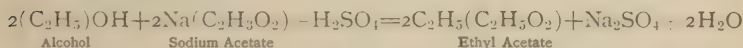
216. Preparation of Nitrous Ether. In a generator mix 1 c.c. of H_2SO_4 and 2 c.c. of HNO_3 . Cool the mixture thoroughly and add 10 c.c. of alcohol. Add a few pieces of broken glass. Boil gently, and collect the distillate in a test tube cooled by ice water. Note the odor of ethyl nitrite or nitrous ether $(\text{C}_2\text{H}_5)\text{NO}_2$. A solution of this in alcohol forms sweet spirits of nitre.

Reaction—



217. Preparation of Acetic Ether. In a generator mix 2 g. of dry sodium acetate, 10 c.c. of alcohol and 2 c.c. of H_2SO_4 . Add a few pieces of broken glass. Heat gently, and collect the distillate in a test tube surrounded by ice and water. Note the odor of ethyl acetate or acetic ether, $(\text{C}_2\text{H}_5)(\text{C}_2\text{H}_3\text{O}_2)$, mixed with some alcohol.

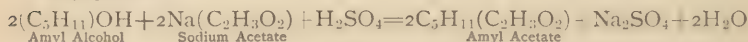
Reaction—



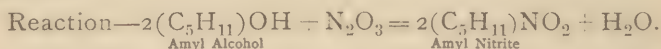
218. Preparation of Amyl Acetate. In a generator mix 2 g. of dry sodium acetate, 5 c.c. of amyl alcohol and 2 c.c. of

H_2SO_4 . Add a few pieces of broken glass. Heat gently, and collect the distillate in a test tube cooled by ice and water. Note the odor of the distillate or amyl acetate, $\text{C}_5\text{H}_{11}(\text{C}_2\text{H}_3\text{O}_2)$, like pears. It is an example of the fruit essences, and is used by confectioners.

Reaction—

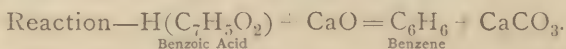


219. Preparation of Amyl Nitrite. Put 3 c.c. of amyl alcohol in a test tube. Through this pass red nitrous fumes of N_2O_3 from a generator containing hot HNO_3 and starch. Keep the alcohol cool by immersing it in ice water. Impure amyl nitrite is formed $(\text{C}_5\text{H}_{11})\text{NO}_2$. Note its characteristic choking odor.



220. Preparation of Benzoic Acid. Place in a dry test tube a small piece of gum benzoin. Gently heat the tube, and note the small white needles of benzoic acid which sublime on the cool part of the tube.

221. Preparation of Benzene. Place in a test tube an intimate mixture of equal parts of benzoic acid and quicklime, and apply heat. Note the odor of the benzene vapor evolved, and compare it with the commercial article. On a larger scale these vapors may be easily condensed.



222. Preparation of Nitro-benzene. In a large test tube mix 10 c.c. of H_2SO_4 and 5 c.c. of HNO_3 . Cool the mixture, add a drop of benzene, shake, and cool the tube. Continue this process until 20 or 30 drops are added, keeping the tube cold. Next slowly pour the whole into a beaker of water. Impure

nitro-benzene, $C_6H_5NO_2$, will sink to the bottom in the form of a brownish yellow oil, called the "essence of mirbane," or, from its odor, artificial oil of bitter almonds.



223. Preparation of Aniline from Acetanilid. Powder a piece of solid NaOH the size of a grain of corn. Mix this with about 1 g. of acetanilid and put it into a test tube. Apply heat. Notice the oily globules that arise as the mixture melts. Sudden solidification will occur. Then tip up the test tube so that the aniline oil may run out. Catch it in another test tube containing about 3 c.c. of water. Keep heating the solidified substance as long as the oil continues to drip from the tube. In the second tube a good-sized globule of white aniline oil, $C_6H_5NH_2$, is obtained. Note the odor, and compare it with that of the commercial article.

Reaction—



224. Color Reaction of Aniline. Take half a test tube of water. Into it put a drop of aniline prepared in the above experiment. Shake well and add 3 drops of a clear solution of bleaching powder in water. A violet color develops, due to the formation of an aniline dye.

225. Preparation of Rosaniline. Take about 2 g. of $HgCl_2$ in a test tube. Add 3 drops of aniline oil. Heat gently until the mass assumes first a green and then a dark purple hue. Cool the tube, and add a little alcohol and two drops of HCl. Stir it up and pour it into a beaker of water. The purple color resulting is due to the presence of rosaniline hydrochloride, one of the most important aniline dyes.

226. Tests for Carbolic Acid or Phenol, C_6H_5OH .

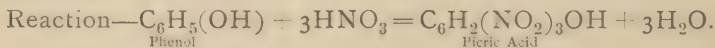
1. *The Ferric Chloride Test.* $FeCl_3$ colors moderately concentrated aqueous solutions dark violet.

2. *The Bromine Test.* A drop of bromine, even in very dilute solutions, ppts. yellowish white tri-bromo-phenol, $C_6H_2Br_3(OH)$.

3. *The Hypochlorite Test.* A pinch of bleaching powder added to an ammoniacal solution of phenol yields a green coloration.

4. *Certain Pine Shavings* moistened with HCl , and touched with a solution of phenol, turn blue when exposed to the air for a time.

227. Preparation of Picric Acid. Take 5 c.c. of dilute HNO_3 in a beaker. Add 2 c.c. of carbolic acid. Cool the mixture. Add 10 c.c. of strong HNO_3 , drop by drop. Boil the solution for five minutes, adding a little strong HNO_3 , drop by drop, if the dark, oily phenol seems to float after some boiling. Pour the whole solution finally into 25 c.c. of water. Set aside and decant the liquid from the yellow crystals deposited. Wash these with a few drops of water. Decant, and notice the yellow solution of picric acid. Dry the crystalline picric acid, $C_6H_2(NO_2)_3(OH)$. Notice that these dry crystals explode when dropped on a very hot surface or introduced into a flame.

**228. Tests for Antipyrin.**

1. *The Ferric Chloride Test.* $FeCl_3$ turns solutions of antipyrin blood red.

2. *The Nitrous Acid Test.* Dilute HNO_3 , having dissolved in it a little KNO_2 , turns solutions of antipyrin green.

3. *The Iodine Test.* I dissolved in KI solution forms, in solutions of antipyrin, a brick-red ppt.

LABORATORY QUESTIONS.

1. Why is sweet spirits of nitre apt to be acid?
2. How might nitrous acid be present in sweet spirits of nitre? If possible, test for its presence by 93, and write the reactions showing its formation.
3. Why would old sweet spirits of nitre turn green on addition of antipyrin?
4. To what class of chemical compounds do fruit essences belong? Do they occur in nature?
5. Ethyl butyrate is pineapple flavor. Write its formula, and invent a method for its preparation. See 237.
6. For what is benzene used?
7. What is the difference between benzene and benzine?
8. Of what two substances is acetanilid composed? Outline a method of preparing acetanilid.
9. For what is amyl nitrite valuable?
10. What compounds of benzoic acid are used in medicine?
11. Why is H_2SO_4 used in the preparation of nitro-benzene?
12. Of what is smokeless powder composed?
13. Why does HNO_3 turn the skin yellow?
14. Is nitro-benzene poisonous?
15. Is carbolic acid an acid? Try litmus paper.
16. What is a carbolate?
17. What is the significance of the termination "ol" in phenol.
18. Is aniline poisonous?

FATS AND OILS.

229. Solution of Fats. Dissolve a small amount of the different oils and fats found on the side table in small amounts of the following solvents: Boiling alcohol, benzol, gasoline carbon disulphide, ether, etc.

230. Emulsion of Fats. Shake a few drops of fat with half a test tube of water. Note that on standing the oil rapidly rises to the surface. Repeat the process with

1. A few drops of alkali. A white emulsion is formed.

4. $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ ppts. insoluble white lead soap (lead plaster).

5. FeCl_3 ppts. insoluble brown ferric soap.

6. FeSO_4 ppts. insoluble greenish ferrous soap.

7. CuSO_4 ppts. insoluble blue copper soap.

234. Preparation of Lead Plaster. Heat in an evaporating dish, with constant stirring, 3 parts of olive oil, 5 parts of water, and 1 part of litharge, PbO , until a yellowish white, tenacious mass of lead soap is obtained. It is the same substance formed in 233-4.

235. Preparation of Fatty Acids. Boil in an evaporating dish a mixture of soft soap and water. Add HCl to excess. Reheat and set aside. Remove the light, fatty substance from the surface. Wash it in water. It is neither fat nor soap. It has no soapy taste. It is a mixture of fatty acids used in the manufacture of candles. Glycerine, KCl and HCl remain in the solution.

236. Preparation of Butter Soap. Put a teaspoonful of butter in a beaker. Boil it with a little dilute aqueous solution of KOH , adding repeatedly small amounts of alkali of increasing strength. After boiling and stirring for some time, remove from the fire and examine the soft butter soap. It will produce a lather.

237. Separation and Identification of Butyric Acid. Mix the butter soap formed above with a little water. Place the mixture in a generator. Add 10 c.c. of H_2SO_4 . The fatty acid may be seen rising to the top. Heat, and collect 5 or 10 c.c. of the distillate in a cooled tube. It will contain butyric acid, have a rancid odor and an acid reaction to litmus paper. Heat this distillate with a little alcohol and 2 c.c. of H_2SO_4 . Note the odor of ethyl butyrate, C_2H_5-

($C_4H_7O_2$), which is used as artificial pineapple flavor. "Oleo" treated thus contains only traces of volatile fatty acids in the distillate, and yields no ethyl butyrate.

238. Identification of Butter and "Oleo." The principle of the above reaction may be more rapidly applied thus: Place in two test tubes equal quantities of butter and oleo-margarine. Add 5 c.c. of a strong alcoholic solution of KOH to each. Warm gently, and notice the difference in odor. The "oleo" has the odor of alcohol simply, the butter the odor of pineapple, by the formation of ethyl butyrate from the alcohol and butyric acid. "Oleo" which has been churned with milk in the process of manufacture may yield slight traces of butyric ether.

239. Decomposition of Glycerine. Mix a few drops of glycerine and H_2SO_4 in a test tube. Heat, and note the sharp odor of acrolein. Any fat containing glycerine yields the same reaction.



240. The Borax Test for Glycerine. Mix with a pinch of borax a few drops of suspected glycerine, which must be neutral and free from ammonium salts. Heated on a platinum wire, the mixture immediately tinges the Bunsen flame pale green, due to the liberation of boric acid by the glycerine.

LABORATORY QUESTIONS.

1. Can castor oil be dissolved in benzene?
2. Can olive, castor and cod-liver oils be distinguished by being touched with H_2SO_4 ?
3. Why does an alkali emulsify fats?
4. Why does albumin water emulsify fats?
5. Does the character of the fat have any influence on the hardness of soap?

6. Why does an alcoholic solution of an alkali more quickly saponify fats than an aqueous solution?
7. Write the reaction involved in the formation of a calcium soap.
8. Why does hard water not lather soap?
9. Why, when an alkali is added to water, does it often become soft?
10. Why is one soap hard and another soft?
11. Is soap a salt?
12. Why is NaCl often added to a kettle of soap after it is made?
13. What lye is usually used in preparing "home-made" soaps?
14. Resin contains 3 fatty acids. Would it saponify with NaOH?
15. What is lead plaster?
16. Which are more stable, the fats of "oleo" or butter?
17. Write the reaction involved in the preparation of fatty acids.
18. In what is oleomargarine superior to butter?
19. Chemically, what is a fat?
20. What is glycerol? What does the termination "ol" signify?

CARBOHYDRATES.

241. Preparation of Corn Starch. Finely pulverize some corn in a mortar. Add water from time to time, and strain the milky liquid off through a cloth. Set it aside until the white sediment can be separated by decantation. Collect and dry this white corn starch, $(C_6H_{10}O_5)_n$, on filter paper.

242. Appearance of Starch Granules. Examine under the microscope a thin section of potato. Notice how the starch granules lie packed within each cell. Touch the section with a drop of dilute iodine solution. Note that the granules turn blue, but the cellulose wall appears unchanged or slightly yellow. Examine the prepared microscopical specimens of potato, arrow root, corn, rice, and other starches, noting the difference in form and size of the granules.

243. Preparation of Starch Paste. Mix a little powdered starch with water to a thin milk. Pour this slowly into a beaker of boiling water with constant stirring. The milki-

ness disappears, and the whole forms apparently a translucent homogeneous solution.

244. The Iodine Test for Starch. A few drops of a solution containing free iodine strike with starch paste a deep blue color, from the formation of starch iodide. This color disappears on heating and reappears on cooling, unless the heat has been high enough to volatilize the iodine.

245. Properties of Glucose. Examine commercial glucose, or grape sugar (dextrose). It is not as sweet as cane sugar. Make a solution of glucose, and set it aside for further experiments. Add H_2SO_4 to cold solutions of cane sugar and grape sugar. Heat to boiling, and note the charring of the cane sugar, while the glucose remains nearly unaltered. Repeat the experiment, using KOH. The cane sugar is only slightly affected, while the glucose assumes a dark brown color, due to the formation of caramel.

246. Fehling's Test for Glucose.¹ In a test tube take some Fehling's glycerine solution. Heat it just to boiling, and add a few drops of liquid containing glucose. Again bring the mixture to the boiling point, and set it aside. If glucose was added in any appreciable quantity, a yellow or brick-red ppt. of cuprous oxide, Cu_2O , will be found in the bottom of the tube. When sufficient glucose is added the copper will all be thrown down and the supernatant liquid will be colorless.

247. Detection of Glucose in Fruits and Candies. Soak a bruised raisin in water and test the solution for glucose. Dissolve a bit of candy in water and test the solution for glucose. Glucose will be present in both instances.

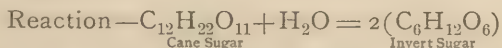
248. Action of Various Sugars on Fehling's Solution. Examine

NOTE 1. The composition of Fehling's glycerine test will be found under 348, Note 1, also other tests for glucose under urinalysis, 346-353.

the following sugars. Make a solution of each, and test as for glucose by Fehling's test. Verify the following conclusions :

- (1) Sucrose (cane sugar). No reducing power.
- (2) Dextrose (grape sugar). Reducing power.
- (3) Lactose (milk sugar). Reducing power.
- (4) Maltose (malt sugar). Reducing power.

249. Preparation of Invert Sugar. Make a dilute solution of cane sugar. Add a few drops of acid, and warm for 10 minutes. Test some of this solution for glucose. Cane sugar takes up a molecule of water and splits into two molecules, one of which is dextrose, or dextro-rotary, the other laevulose, or laevo-rotary.



250. Action of Sugar on Polarized Light. Examine with the polariscope a solution of cane, malt, milk, or grape sugar. Note that they are dextro-rotary. Examine a solution of invert sugar, and decide on its action on the polarized ray.

251. Preparation of Barley Sugar and Caramel. In an evaporating dish very slowly heat some dry cane sugar. It melts to a clear yellowish liquid. When melted, stir and cool. Barley sugar is formed. Save a sample. Heat the remainder until it is of a dark brown color. Caramel is formed, which is sugar less a molecule or two of water. It is used for coloring liquors, etc. Test solutions of both these products for glucose, and prove its presence.

252. Conversion of Starch to Dextrine. Heat a pinch of dry, finely powdered starch in an evaporating dish over a *very slow* fire, with continuous stirring until it *all* turns brown, but does not char. This occurs at about 250° C. Dextrine or British

gum is formed, $C_6H_{10}O_5$. When cool, boil with a little water, and filter. The solution should be of a reddish color, and if all starch has been transformed to dextrine, the solution strikes a pinkish and not a blue color with iodine.

This is an example of the splitting of the starch molecule into soluble starches or dextrines, which action is induced by heat, acids and digestive ferments. It may be continued through a series of products, ending at last in the formation of sugar, as seen in the next exercise, and in 282, under Saliva, where the color reactions of the intermediate products with iodine are given.

253. Conversion of Starch to Glucose. Boil a beaker of starch paste, prepared as in 243, with 10 c.c. of H_2SO_4 for an hour or more, adding water from time to time, if necessary, until a drop of it ceases to give a color with iodine, showing the conversion of all the starch. Add marble dust until effervescence ceases, which neutralizes any free acid remaining. Filter the solution. Notice its sweet taste. Test it, and prove the presence of glucose. It may be evaporated, and will sometimes crystallize. This is the method of preparing commercial glucose from corn, and is much used in syrups, candies, etc.

254. Conversion of Starch to Maltose. In a test tube warm to blood heat a dilute solution of starch paste. Pound up some malted barley—*i. e.*, barley which has begun to sprout, and dried and heated until the germ is killed. Mix this with water. Grind it up and filter. Add some of this clear solution to the warm starch paste. After some minutes, test and prove the presence of maltose by Fehling's test. Prove the conversion of all the starch by a drop of iodine solution.

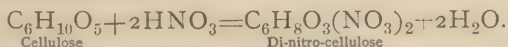
Illustrative reaction— $(C_6H_{10}O_5)_3 \cdot 2H_2O = C_{12}H_{22}O_{11} + C_6H_{12}O_6$
Starch Maltose Dextrose

255. Preparation of Parchment Paper. Dip a piece of unsized

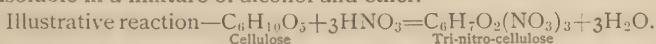
paper in strong H_2SO_4 for about 15 seconds, then wash it thoroughly with water. Notice that the paper has become stronger and more translucent. Its fibers have become colloidal, and it may be used for dialysis, as it will allow only the molecules of crystalline substances to penetrate its pores, and the simpler proteid molecules, like peptones, etc.

256. Preparation of Collodion. Make a mixture of 15 c.c. of strong H_2SO_4 and 20 c.c. of strong HNO_3 in a beaker. Cool the mixture and immerse in it a piece of shredded absorbent cotton. Macerate 15 minutes. Wash the cotton free from acids under the tap. Press it between filter paper and dry it. Notice that when thoroughly dry a portion burns instantly, leaving little ash. If the nitration has been exactly performed, di-nitro-cellulose is produced. This is pyroxylin, or soluble cotton. It dissolves in a mixture of 3 parts of ether and 1 of alcohol, forming collodion.

Illustrative reaction—



Gun-cotton is more explosive, and consists of a mixture of higher nitrates of cellulose, produced by more prolonged action of HNO_3 . It is insoluble in a mixture of alcohol and ether.



LABORATORY QUESTIONS.

1. Can the source of starch be determined by the form of its granules?
2. Write the formulas and show the relations of cellulose, starch and the various sugars.
3. What is a carbohydrate?
4. What is the nature of the change when cane sugar becomes glucose?
5. Why is vinegar, cream of tartar, or lemon juice used in candy making?
6. In how many ways may starch be converted into glucose?
7. Ought glucose to be unhealthful?

8. For what is dextrine used?
9. How does a potato sprout bring its insoluble starch up to its leaves?
10. Why is maple sap sweet in the spring? Why does it lose its sweetness when the leaves start?
11. What does malt extract contain? Is its nutritive value diminished by fermentation?
12. Could saw-dust be made available for food?
13. Why do fruits sweeten as they ripen, and lose their sweetness when they decay?
14. Why is invert sugar so called?
15. What is malt?

LABORATORY EXERCISES
IN
PHYSIOLOGICAL CHEMISTRY
INCLUDING THE
DIGESTION, BLOOD AND MILK

PHYSIOLOGICAL CHEMISTRY.

THE DETECTION OF PROTEIDS IN SOLUTION.

257. The Xanthoproteic Color Reaction. Take half a test tube of albumin solution. Add 5 c.c. of concentrated HNO_3 . Warm, and notice the yellow precipitated albumin. Divide in two test tubes. Add, until alkaline, to the one NH_4OH , to the other KOH . The yellow solution turns orange in both instances.

258. The Biuret Color Reaction.¹ Take half a test tube of albumin solution. Add two drops of *dilute* CuSO_4 and 5 c.c. of KOH . A violet tint appears, which deepens on boiling. With peptones the color is rose-red.

259. Millon's Precipitation Reaction. Take half a test tube of albumin solution. Add a few drops of Millon's reagent.² A white ppt. falls, becoming reddish on boiling. If only traces of albumin are present, the solution becomes slightly red.

260. The Sodium Sulphate Precipitation Reaction. Take one-third of a test tube of albumin solution. Render it acid with acetic acid. Add an equal volume of concentrated Na_2SO_4 solution. When boiled, a white ppt. falls. This test throws down all proteids except peptones. The salt and acid do not interfere with the tests for peptones in the solution.

261. The Ferrocyanide Precipitation Reaction. Render a

NOTE 1. Called Biuret because this color also forms from biuret, $\text{C}_2\text{H}_5\text{N}_3\text{O}_2$, a substance obtained by heating urea.

NOTE 2. Millon's reagent is prepared by dissolving 1 part of metallic mercury in 2 parts of strong HNO_3 , adding twice its volume of distilled water, setting aside until settled, and decanting the clear solution.

solution of albumin strongly acid with acetic acid. Add a few drops of a fresh, strong K_4FeCy_6 solution. A milk-white precipitate falls. This reagent ppts. all proteids except peptones and some forms of albumose. When acetic acid is added after the K_4FeCy_6 , mucin is not precipitated.

262. The Tannic Acid Precipitation Reaction. Take half a test tube of albumin solution. Add 1 c.c. of a strong solution of tannin. A white precipitate falls. Tannic acid reacts with all proteids.

263. The Picric Acid Precipitation Reaction. Take half a test tube of albumin solution. Add a few drops of a strong solution of picric acid. A yellow ppt. comes to view, dissolving to a dark color when heated with KOH. Picric acid ppts. all proteids.

264. The Absolute Alcohol Precipitation Reaction. Take one-third of a test tube of albumin solution. Render it acid with acetic acid. Add an excess of absolute alcohol. A ppt. falls. This reagent precipitates all proteids, including peptones.

EXAMINATION OF THE VARIOUS PROTEIDS.

CLASS I.—ALBUMINS.

Albumins are proteids soluble in water and coagulated by heat.

265. Preparation of Egg Albumin. Take an egg. Break a small hole in one end of the shell. Pour the white into a beaker, leaving the yolk for future examination. Half fill the beaker with water. Stir with a glass rod, breaking up the albumin thoroughly. Filter through a piece of muslin, and keep the solution for the following reactions.

266. Precipitation of Albumin by Heat. Boil half a test tube

of albumin water. Notice the coagulation. The coagulum does not dissolve on addition of HNO_3 .

267. Temperature of Coagulation. Extract a little more of the undiluted white of egg from the shell. Place it in a test tube, insert a thermometer, and set the whole in a beaker of water. Warm the beaker slowly. Note the temperature at which the albumin shows the first signs of opalescence (about 59°C.), and the point of total coagulation (about 73°C.).

268. Precipitation of Metallic Albuminates. Prepare three test tubes of albumin solution. Add to separate tubes solutions of CuSO_4 , AgNO_3 , and HgCl_2 . Copper, silver and mercury albuminates fall. Compare them with the soap ppts. formed in 233.

CLASS II.—PEPTONES.

Peptones are proteids soluble in water, but not coagulated by heat. Albumoses are substances between albumins and peptones.

269. Action of Heat and Reagents. Using the prepared solution of peptones, confirm the following reactions:

1. Not coagulated by heat.
2. Not pptd. by adding NaCl .
3. Not pptd. by acids or alkalies.
4. Not pptd. by Na_2SO_4 , as in 260.
5. Not pptd. by K_4FeCy_6 , as in 261.
6. Pptd. by tannic acid, as in 262.
7. Pptd. by absolute alcohol, as in 264.
8. Yields the rose-red Biuret reaction, 258.

CLASS III.—GLOBULINS.

Globulins are proteids insoluble in water, soluble in dilute NaCl . Solutions are coagulated by heat. Among the most important are:

Vitellin, crystallin (globulin), myosin, fibrinoplastin (paraglobulin), and fibrinogen (metaglobulin).

270. Preparation and Detection of Vitellin. Stir up the yolk of the egg saved, after washing it as carefully as possible from the white. Put 3 c.c. of yolk in a test tube. Shake well with half a test tube of ether several times. Allow the yellowish ether to rise each time, and pour it into an evaporating dish. The ether largely dissolves the fatty matters. Impure vitellin remains. Very gently warm the tube, and set it aside until the smell of ether disappears. Add water. The vitellin does not dissolve. Next add a pinch of NaCl. Vitellin dissolves to a milky solution. Filter, and apply the Heat (266), the Xanthoproteic (257), and the Biuret (258), reactions.

271. Examination of the Fatty Matter of the Yolk. Evaporate the ether solution in the evaporating dish very cautiously, lest the ether take fire. In case it should, cover the dish quickly with a damp cloth, which will extinguish the flame. A yellow oil is left. Pour a few drops into water. Globules of fat are formed. Heat the remainder with a few drops of HNO_3 . The solution is first colored blue, then fades to green, and becomes colorless. Now add a little water and a drop of KCyS solution. A reddish color proves the presence of iron.

272. Preparation and Detection of Crystallin. Extract the crystalline lens from the eye of an ox. Thoroughly grind it up in a mortar with 10 c.c. of water, which will dissolve albumin. Filter, and grind the residue with 10 c.c. of a 10% salt solution. The crystallin dissolves. Filter, and confirm its presence by Millon's test (259), the Biuret test (258), and the Heat test (266).

273. Preparation and Detection of Myosin. Soak a table-

spoonful of finely chopped lean meat in a beaker of water for 10 minutes. Squeeze out the juice through a wet cloth, and test the liquid for albumin by Millon's reaction (259.) Wash the cloth. Repeat the digestion several times, until the water no longer yields much, if any, ppt. with Millon's reagent, showing the removal of serum albumin. Now soak the washed meat in a 10% salt solution. Strain the liquid. Filter, and test for myosin by Millon's reagent and a few other tests for proteids (257-264).

Fibrinogen and Fibrinoplastin (310), may be similarly prepared, but the process is too tedious for the time allowed.

CLASS IV.—DERIVED ALBUMINS.

Derived albumins are proteids insoluble in water, soluble in dilute acids or alkalis. Solutions not coagulated by heat.

274. Preparation of Acid Albumin. Take half the white of an egg. Add 10 drops of glacial acetic acid. Whip them well together for some minutes until a gelatinous mass of acid albumin forms. Dissolve this in half a beaker of warm water. Filter, and confirm the following reactions for solutions of acid albumin:

1. Not coagulated by heat.
2. Precipitated when *exactly* neutralized by NaOH.
3. This last ppt. is dissolved by an excess of alkali.

275. Preparation of Alkali Albumin. Take half the white of an egg. Add 15 drops of strong KOH solution. Whip them well together until a gelatinous mass of alkali albumin is formed. Dissolve this in warm water. Filter, and confirm the following reactions for solutions of alkali albumin:

1. Not coagulated by heat.
2. Precipitated when *exactly* neutralized by HCl.
3. This last ppt. is dissolved by an excess of acid.

276. Preparation of Syntonin (muscle acid albumin). Wash a tablespoonful of lean chopped muscle until proved by Millon's reagent to be free from serum albumin, as in 273. Digest the washed meat in a 0.2% HCl solution. Strain the solution through cloth. Filter, and confirm the following reactions for solutions of syntonin:

1. Not coagulated by heat.
2. Precipitated when *exactly* neutralized by NaOH.
3. Yields the Millon's and Biuret reactions.

Casein, sometimes classed as an alkali albumin, will be considered under Milk, 320.

CLASS V.—FIBRIN.

Fibrin is a proteid insoluble in water, swollen by salt solutions, slowly soluble in dilute acids and alkalis. Solutions are coagulated by heat.

Fibrin will be examined under Blood, 305.

CLASS VI.—COAGULATED PROTEIDS.

Proteids are insoluble in water; insoluble in dilute acids or alkalis, but decomposed by them when heated.

277. Preparation and Reaction of Coagulated Proteids. Coagulate some albumin solution by heat. Use portions of this coagulum to confirm the following reactions.

1. It is insoluble in water.
2. It yields the Xanthoproteic, Biuret and Millon's reaction.
3. It is changed to acid and alkali albumin by heating with acids and alkalis.

CLASS VII.—LARDACEIN.

Lardaccin, or amyloid substance, is a proteid insoluble in water, salt solution, dilute acids or alkalis, and unacted upon by digestive ferments. Iodine stains it reddish brown.

CLASS VIII.—ALBUMINOIDS.

Albuminoids are substances containing N, related to proteids, but not answering all proteid reactions, and yielding different decomposition products. Among the principal albuminoids are collagen (ossein), chondrin, elastin, gelatin, keratin, nuclein, mucin and spongin.

LABORATORY QUESTIONS.

1. What effect does heating have on protoplasm?
2. What is an albumose?
3. What is lardacein?
4. What is an albuminoid?
5. What is glue? Is it a proteid? To what class does it belong?
6. What proteids could a neutral, boiling meat extract contain?
7. Does beef tea contain starch or sugar? Test, if possible.
8. Why are not peptones suitable for food? Taste an artificial digestion, and see.
9. If some proteids were dissolved by ice water, what would be the effect of warming the soup?
10. Would salt water extract more nutritive properties from beef than fresh water?
11. Name a wide-spread metallic compound of a proteid. Where have you dealt with it under the head of proteids?
12. In testing urine for albumin, would it be wise to select a test reacting for all proteids?
13. Select from the general tests for proteids the one best adapted to detect serum albumin, and that only, in the urine.
14. What forms of albumin would dialyze in the stomach, and what forms in the intestines?
15. Why is the blood advantageously saline and alkaline?
16. What is a nucleo-albumin?

SALIVA.

278. Detection of Sulphocyanides in Saliva. Put a clear drop of saliva on a white porcelain surface. Touch this with a drop of dilute FeCl_3 . A light claret color is *usually* produced, especially on standing, which is not destroyed by a

solution of HgCl_2 . This test proves the presence of a salt of sulphocyanic acid, HCyS .

279. Action of Ptyalin on Starch. Prepare a dilute solution of starch paste, 243, and collect a few c.c. of saliva. Test both by Fehling's solution, and prove that neither contains glucose. Mix the two, warm to blood heat for 10 minutes, after which prove the presence of glucose by Fehling's test.

280. Reaction of Saliva. Into a few c.c. of clear filtered saliva put a few drops of violet litmus solution. Compare it with a similar test made with water. If the saliva is alkaline, a deeper hue results with the saliva. In rare cases the action may be acid. It is most alkaline at the height of the flow, amounting to about that of a .08% Na_2CO_3 solution.

281. Inactivity of Ptyalin in Acid Solution. Repeat experiment 279, rendering the starch slightly acid with acetic acid before adding the saliva. At the expiration of 10 minutes Fehling's test shows little or no glucose, which points to the inactivity of ptyalin in acid solutions.

282. Determination of the Diastatic Value of Saliva.¹ Take 10 c.c. of the standard starch solution,² in a beaker, and dilute with 90 c.c. of water. Warm to 40°C . Add 1 c.c. of clear, carefully-measured saliva.³ The whole is kept stirred. At the end of every minute a drop is removed to a white porcelain surface and touched with a drop of dilute iodine solution.

NOTE 1. This method may be applied to the determination of the diastatic value of infusions of malt or diastase, etc.

NOTE 2. The standard starch solution is prepared by taking 10g. of starch, making it into starch paste, and diluting with water until the whole measures 1,000 c.c. This solution must be used when fresh, and well shaken. Each c.c. contains .01 g. of starch.

NOTE 3. The experiment must be tried several times, and the amount of saliva used diminished or increased until the achromic point is reached within 4-6 minutes, otherwise the results are not reliable.

The blue color at first formed passes through violet, pink, brown and yellow, and at last reaches the colorless or achromic point, when the conversion of the starch has completely passed through amylo-, erythro-, achroo- and malto-dextrin to maltose.

RESULT OF AN ACTUAL EXPERIMENT.

10 c.c. of starch, 90 c.c. of water, 0.2 c.c. of saliva, temp. 40° C.

Time.		Reaction with Iodine.
6 minutes.	4.15 P. M	Begun.
	4.16 "	Blue.
	4.17 "	Violet.
	4.18 "	Brown.
	4.19 "	Yellowish brown.
	4.20 "	Pale yellow.
	4.21 "	No color, achromic point.

The diastatic value may then be calculated, showing how much starch solution 1 c.c. of saliva will change in 5 minutes at the temperature of the experiment. Thus:

The diastatic value = $\frac{10}{\text{No. of c.c. of saliva}} \times \frac{5}{\text{No. of minutes}}$
 or in the example $\frac{10}{.2} \times \frac{5}{6} = 41.6 \text{ c.c.}$

LABORATORY QUESTIONS.

1. Under what circumstances might saliva be acid?
2. Does the presence of cyanides in saliva seem to indicate the breaking down of proteids to supply the salivary secretion?
3. How much dry starch would 1 c.c. of the saliva in the example change into maltose in 5 minutes?
4. What seems to be the chief use of saliva?
5. Is the activity permanently destroyed by rendering ptyalin acid? Neutralize the acid in experiment 281, and see if the ptyalin resumes its amylolytic activity.
6. Will saliva answer the tests for proteids? If so, to what is this principally due?
7. What evidence do the teeth show that the saliva contains inorganic salts?

8. If possible, dry some saliva. Ignite the residue, and test it for chlorides, carbonates and phosphates.

9. Are medicinal substances excreted in saliva? See 293.

10. Most enzymes are soluble in glycerine. Would a pig's salivary gland, pounded and soaked in glycerine, yield an active amylolytic solution? If possible, try it.

11. Is any student found whose saliva does not contain ptyalin? If so, examine his saliva at different times in the day. Assign a cause, based on health, constitution and habits.

12. What influence would tea have on pepsin and enzymes in general?

13. Why does saliva bubble?

14. What is the cause of the alkalinity of the saliva?

GASTRIC JUICE.

283. Dialysis of Gastric Digestion. Make a dialyzer by firmly tying with a thread a piece of fresh, thin sausage skin over the mouth of a large bottomless test tube. Half fill this with the liquid product of an artificial digestion, containing digested and undigested albumin in solution. Immerse the dialyzer in a beaker of water. After some hours confirm the following reactions :

Test Employed.	Contents of Beaker.	Contents of Dialyzer.
Heat 266	Not coagulated.	Coagulated.
Biuret 258	Pink.	Violet.
Ferrocyanide . . . 261	Not precipitated.	Precipitated.
Millon's 259	Precipitated.	Precipitated.

284. Preparation of an Active Pepsin Solution. Open the fresh stomach of a pig and very gently sponge it off. With a sharp knife scrape off the mucous membrane, containing the peptic glands. Put this, finely minced, in a beaker and cover with five times its volume of water, acidulated so as to contain 0.1-0.2% of HCl. Keep it at a temperature of about 40° C, and after a few hours nearly all will have disappeared.

Filter and preserve. The solution will possess active proteolytic properties, and keep well.

The liquid might be subjected to dialysis to remove most of the peptones, then the enzyme with some proteid matter precipitated by absolute alcohol, dried and preserved as "pepsin."

285. Artificial Gastric Digestion. Into a dialyzer, prepared as in 283, put some fresh, well-washed fibrin clots or finely diced white of a hard-boiled egg. Acidulate a beaker of water so that it may contain 0.1–0.3% of HCl. Add some of this solution to the dialyzer, and immerse it in the beaker of acidulated water. Drop a little pepsin or some of an active proteolytic extract of a pig's stomach into the dialyzer. Keep the whole at a temperature of about 40° C, and agitate as frequently as is convenient. Note how the fibrin swells before solution. After some hours, test the contents of the beaker for peptones by the Biuret and Millon's reactions. Note the bitter taste of the solution always present with peptones.

286. The Action of Rennin. Carefully neutralize a little of the active extract of the pig's stomach, and add it to a test tube of warm milk. If rennin is present, milk is coagulated in a few minutes.

Or, soak a piece of cat's stomach or true rennet, calf's stomach, in dilute HCl, or in a strong solution of salt, for 10 minutes. Exactly neutralize the solution, if necessary, with NaOH, and add it to warm milk. The solution soon coagulates.

CHEMICAL EXAMINATION OF THE GASTRIC JUICE.

287. Collection of the Juice by Ewald's Test Breakfast. When the stomach is empty, administer a roll weighing about

35 g. (540 grains), with about 300 c.c. (10 f $\frac{3}{4}$) of warm water or tea without sugar or milk. After one hour, remove the remnants of the meal by the use of the stomach tube. About 40 c.c. (11 f $\frac{3}{4}$) should be obtained. A variation of 20 c.c. either way would be pathological.

288. Detection of Free Acids. Add a few drops of gastric juice to a dilute yellowish red solution of *tropaeolin*. If free acids are present, even in as small a proportion as 1-4,000, the color instantly changes to reddish brown. Acid salts turn the solution yellow. *Congo red* may be used likewise. If free acids exist, even in as small a proportion as 1-50,000, the red color of the solution instantly turns sky blue. Both organic and inorganic acids affect these reagents.

289. Estimation of Total Acidity. 10 c.c. of clear filtered juice, diluted with about 30 c.c. of water, are placed in a beaker, a few drops of neutral phenol-phthalein added, and the whole titrated with $\frac{n}{10}$ NaOH from a burette. The alkali is added drop by drop, with constant stirring, until a faint pink becomes permanent. This usually requires about 5 c.c. of alkali. The number of c.c. used, multiplied by .0364 = % of acid, were all the acid HCl.

290. Estimation of Free Acids. 10 c.c. of clear filtered juice is diluted with 30 c.c. of water, treated with 1 g. of pure CaCO_3 (precipitated chalk), which is neutral itself and neutralizes free acids but not acid salts. When effervescence ceases, the whole is titrated with $\frac{n}{10}$ NaOH, as above. The amount of alkali used measures the acid salts. The difference between this and the previous determination represents the free acids.

291. Günzburg's Color Test for Free HCl. A few drops of gastric juice are placed in a clean white evaporating dish and

gently heated. A glass rod is dipped in Günzburg's reagent¹, and drawn through the hot juice. A deep scarlet line is produced, if even 1 in 20,000 parts of free HCl is present. Phloro-glucin-vanillin does not react with organic acids or normal amounts of acid salts.

292. Uffelmann's Test for Lactic Acid. Mix 6 drops of dilute FeCl_3 with 2 drops of carbolic acid, and add water until this test solution is a delicate amethyst blue. Take some of this in a test tube and add a few drops of gastric juice. If lactic acid is present, even in the proportion of 1-2,000 parts, the color instantly becomes yellow. By this test pure gastric juice is proved to contain no lactic acid. Whenever present it is set free from the food.

293. Rapidity of the Stomach's Absorption. Administer a capsule containing 0.1 g. (1.5 gr.) of KI. It will normally appear in the saliva in 10 to 15 minutes. To detect it, touch the tongue every few moments to a slip of starch iodide paper, 57-Note 1, and moisten the spot with a drop of dilute, yellow HNO_3 . A blue coloration shows I. Note the time.

294. Ewald's Test of the Stomach's Motility. Administer in a capsule 1 g. (15 gr.) of salol (phenol salicylate). It is not decomposed until it reaches the alkaline contents of the duodenum. It can be detected normally in about 70 minutes. Urine is voided at short intervals. Paper is moistened in it and touched with a drop of 10% FeCl_3 solution. The edge of the drop assumes a violet color if a trace of salicylic acid is present.

NOTE 1. Günzburg's solution is made by dissolving in 30 c.c. of absolute alcohol 2 g. of phloroglucin and 1 g. of vanillin. The solution is light yellow, with the odor of vanilla.

LABORATORY QUESTIONS.

1. How many important uses has HCl in the gastric secretion?
2. How could syntonin be tested for in exercise 285?
3. Will the active extract of the pyloric end of a pig's stomach digest starch in acid solution? Try it, if possible.
4. Is the stomach ever alkaline, and why?
5. Why does the living stomach not digest itself?
6. Does the acidity of the stomach aid in dialysis?
7. What is the zymogen theory of enzymes?
8. State the theory of the formation of HCl in the stomach?
9. What can be said of the comparative sizes or shapes of the albumin and peptone molecules, as deduced from their diffusibility?
10. State the probable nature of the change when proteids are broken down into albumoses and then to peptones.
11. Why is rennet usually soaked in acid or salt solution?
12. Will gastric juice digest raw white of egg as quickly as cooked? Why?
13. State how the gastric juice assists in the digestion of fats.
14. How does the gastric juice aid in the digestion of sugar?

PANCREATIC JUICE.

295. Preparation of a Solution of Pancreatic Enzymes. Clean a portion of a pig's pancreas as free as possible from fat. Cut it up fine, and crush it in a mortar with pieces of glass. Cover this comminuted material with five or ten times its volume of saturated salt solution. After several days filter off the active extract. This will contain all the pancreatic enzymes except the fat-splitting enzyme. If it is desired to keep the solution, glycerine should be used as a solvent, or a little alcohol added to the salt extract.

296. Action of Trypsin. Place in a dialyzer, 283, a coagulum of fibrin. Prepare a beaker of water containing 1% of Na_2CO_3 . In the dialyzer put a solution of pancreatin or pancreatic extract, prepared as above. Fill the dia-

lyzer with the alkaline solution from the beaker, and set the whole in the beaker containing the remainder of the alkaline liquid. Keep the solution at a temperature of about 40°C , and stir occasionally. The fibrin will gradually disappear without the swelling observed in gastric juice.

297. Action of Rennin. Fill a test tube half full of sweet milk. Add 5 c.c. of pancreatic extract. Place the whole in a beaker of water, and warm to 40°C . In a few moments the milk will coagulate. Later the coagulum dissolves, digested by the trypsin. Confirm the following conclusions:

(1) The solution has the bitter peptone taste.

(2) The casein no longer curdles on boiling or on addition of HNO_3 .

(3) The Biuret test yields a rose-red color.

298. Action of Amylopsin. Fill two test tubes half full of dilute starch paste. Add a little pancreatic extract to each. Render one slightly alkaline with Na_2CO_3 , the other acid with HCl . Warm to the usual temperature. After 5 minutes test each for glucose by Fehling's test. The alkaline solution proves active. The acid solution is inactive.

299. Action of the Fat-Splitting Enzyme. Dissolve a little butter in 5 c.c. of ether. Add a few drops of violet litmus solution. Beside it set a blank test of similarly colored water. The reaction of the butter solution must be *exactly* neutral, or made so, and the color a faint violet. Soak a small piece of fresh pancreas for 10 minutes in strong alcohol, and tease it up well. Remove it. Dry it with a filter paper, and immerse it quickly in the ether solution. After some minutes the litmus shows a decided red tinge. The solution has become acid by the liberation of butyric acid by the enzyme.

LABORATORY QUESTIONS.

1. If fresh juice from a pancreatic fistula is at hand, determine the reaction, color, taste, effervescence with acids, emulsive power with fats, especially acid fats, and the coagulation of the drops of juice when poured into water.

2. How do the products of gastric and pancreatic digestion differ?

3. Are the final products of gastric or pancreatic digestion best suited to enter the blood? Does Nature sustain your conclusion?

4. Will the pancreas digest itself, as did the stomach in 284? Try it, if possible.

5. Which keeps best, a solution of gastric or of pancreatic digestion? Why?

6. Write the reaction when butter is split by an enzyme.

7. Which shows the rennet action more strongly, gastric or pancreatic extract?

8. Are physiologists agreed as to the presence of rennin in the pancreatic fluid?

9. Will a piece of the small intestine curdle milk?

10. Judged by your experiments, which has the greater amylolytic power, pancreatic extract or saliva?

11. How does the digestion of fibrin differ in acid and alkaline digestive fluids?

BILE.

300. Properties of Bile. Notice the reddish yellow color of fresh bile. That of herbivora is usually a greenish yellow. Note its bitter taste, due to bile acids, and its feeble alkaline reaction when diluted. Test its power of emulsifying fats, by shaking with a few drops of oil.

301. Separation of Cholesterin. The presence of cholesterin in bile is best shown from biliary concretions or gall stones. If these are colored, powder one, boil with water several times until the color is removed and the water decants clear. Next boil the clean powder for some minutes with a little alcohol. Set aside for a moment. When settled, decant

some of the clear alcoholic solution on two clean glass microscope slides. Warm these gently, and set aside. When dry, examine with the microscope for the characteristic large, flat, clear crystals of cholesterin. One specimen will turn red when touched with a drop of H_2SO_4 , the other blue with a mixture of H_2SO_4 and tincture of iodine.

302. Gmelin's Test for Bile Pigments.

(1.) Put an inch of clear HNO_3 in a test tube. Add one tiny crystal of cane sugar, warm, and the acid becomes light yellow, due to the presence of a little nitrous acid. When cold, gently let a few drops of dilute bile from a pipette flow upon the surface of the acid. At the juncture of the liquids there will appear colored rings, first green, then blue, violet and red, passing down the acid. Indican will yield only red and violet rings. Lutein yields only green and blue. Occasionally bile does not *immediately* yield these colors.

(2) Mix a solution of bile with a strong solution of KNO_3 . Let a few c.c.'s of concentrated H_2SO_4 flow down the side of the inclined test tube and underlay the solution without mixing with it. The characteristic bile colors, as above, appear at the junction of the liquids.

303. Pettenkofer's Test for Bile Acids. Take half an inch of clear diluted bile in a test tube. Add just 5 drops of a 10% cane sugar solution. Next add H_2SO_4 , drop by drop, cooling the tube thoroughly after the addition of each drop, until nearly an equal quantity of H_2SO_4 has been added. If the tube becomes warm, the sugar becomes slightly carbonized and the test ruined. If bile acids are present, the fluid becomes first opalescent, then clear, and successively a cherry red, carmine-red and purple-violet. Sometimes

these colors require several minutes to appear. Albumin, amyl alcohol, fatty acids, morphine, phenol compounds, etc., give this reaction, so that it cannot be applied to complex organic mixtures without tedious preparation and precaution.

304. Spectroscopical Examination of Bile. Set a thin, flat bottle filled with a clear, dilute solution of bile before the slit of the spectroscope. Examine the absorption spectrum produced, with the black bands at each side, obscuring all but the red and yellow colors. Fix the exact point of termination of these bands by means of the scale in the instrument. They will be found to differ somewhat, according to the dilution of the bile. Place before the slit any other similarly colored solutions not containing bile, and notice that they do not produce the same characteristic absorption bands.

Concerning the adjustment of the instrument, consult the demonstrator.

LABORATORY QUESTIONS.

1. What salt in human bile does Pettenkofer's test principally detect?
2. Do the various colors in Gmelin's reaction probably correspond to definite oxidation products? If possible, examine a changing Gmelin's reaction by placing the test tube before the spectroscope slit.
3. Try Pettenkofer's test with urine containing bile. What is the result?
4. Does bile contain iron? Evaporate and ignite some. Dissolve in dilute hot HNO_3 , dilute and filter. Touch the solution with a drop of KCyS . A red color indicates iron.
5. What are the bile coloring matters and the theory of their origin?
6. Will pig's bile digest *raw* starch? If possible, try it.
7. Will bile dissolve insoluble soaps? Make a calcium soap, 233-1, and confirm it, if there is time.
8. Will bile precipitate solutions of peptones, pepsin, acid albumin, etc.?
9. Will bile reduce cooked starch to sugar? Try it.

BLOOD.

305. Preparation of Fibrin. Stir some freshly-drawn blood with a stirring rod, or better, a bundle of wires, until the fibrin is all deposited around the rod or wires. Wash this in water until white, and prove it a proteid by the Xanthoproteic reaction, 257.

306. Microscopical Appearance of Blood. Place a drop of the defibrinated blood on a microscope slide, cover it with a cover glass, and examine with the high power of the microscope. Detect the red and white corpuscles. Compare the appearance of human blood with that of the dog, cow, bird, fish, etc., by examining the slides prepared.

307. The Odor of Blood. Take some of the defibrinated blood in a test tube. Add a few drops of H_2SO_4 . Stir up the solution, and note the peculiar odor of blood, intensified by the liberation of traces of volatile acids by the H_2SO_4 .

308. Preparation of Reduced Haemoglobin. Make a reducing solution as follows: In a test tube put a strong solution of SnCl_2 , add a little powdered tartaric acid, then NH_4OH until the solution is quite clear and alkaline. This solution will absorb oxygen. Shake in a test tube some blood with a few c.c.'s of this reducing solution. The color changes to the dark purplish red of reduced haemoglobin. Keep agitating the blood in the presence of air, and it gradually resumes the bright red color of oxyhaemoglobin.

309. Tests for Serum Albumin. Pour some serum from a large blood clot. Dilute it with 10 times its volume of water, and use the solution in the following tests:

(1) *The Ferrocyanide Test* (343). Keep diluting a small amount of serum albumin successively with 9 times its vol-

ume of water, thus making a $\frac{1}{10}$, a $\frac{1}{100}$, etc., solution. Test each time, and determine the limit of delicacy.

(2) *The Heat and Acid Test* (341).

(3) *Heller's Nitric Acid Test* (342). Keep diluting the serum albumin, as above, until the limit of detection is reached. Compare the delicacy of this with the K_4FeCy_6 test.

310. Preparation and Detection of Paraglobulin (Fibrinoplastin, or serum globulin). Pass CO_2 , 120, through a beaker of dilute serum for 20 minutes or more. Let the ppt. settle. It is paraglobulin. Decant and, after washing with water, dissolve some of it in a little dilute salt solution. Test it by the Biuret test, 358, and prove it a proteid.

311. Detection of Fat in Blood. In a large test tube shake equal quantities of blood serum and gasoline, which substance will dissolve fat. Let the gasoline rise, and pour it into a watch glass or evaporating dish. When evaporated, examine for globules of fat.

312. Detection of Some Salts of the Blood. Evaporate some blood to dryness in an evaporating dish. Raise the temperature to a red heat. When cool, add a little HNO_3 , heat, dilute and filter. Test some of the solution as follows:

1. Add $KCyS$. A red color indicates iron.
2. Add $(NH_4)_2MoO_4$, and boil. Yellow ppt. on standing indicates phosphates.
3. Add $AgNO_3$. A white cloud indicates chlorides.

313. The Guaiacum Test for Blood. Moisten any dry blood stain and press it against a piece of blotting or filter paper. A faint reddish impression is taken on the paper. Touch this with a drop of a fresh tincture of guaiacum, and then

with one drop of hydrogen dioxide, or, better, "ozonized ether."¹ A blue color rapidly appears. Saliva, protoplasm, semen, milk, etc., give this reaction, but in practice blood is the only red stain which yields it, except fruit stains, which turn blue with ammonia. Red wine stains give this reaction only after standing some hours. The test is valuable in connection with other facts.

314. The Haemin Test for Blood Stains. Cut out a dry blood stain from a piece of cloth, no matter how old the stain. Place it on a glass microscope slide. Add *exactly* 3 crystals of NaCl and 2 drops of glacial acetic acid. Heat gently until it boils, then squeeze the acid from the cloth. Gently evaporate the liquid. When dry, examine the slide, under the high power of the microscope, for the small, dark red bundles, rods or prismatic crystals of hydrochlorate of haemin. They must not be mistaken for the large, transparent crystals of NaCl. In case they do not appear, repeat the process with the same stain until expert. Only blood will yield this test.

315. The Spectroscopic Examination of Blood. Place before the slit of the spectroscope a thin, flat bottle filled with clear blood, diluted with water so that the blood forms only about $\frac{1}{10}$ of 1 per cent. Examine the absorption spectrum, noting the black bands on either side and the two black bands in the center. Fix the position of all these by means of the scale in the instrument. The exact extent of these bands depends upon the dilution of the blood. Now place before the slit, dilute solutions of fuchsin, cochineal, etc., prepared by the

NOTE 1. Ozonized ether is prepared by shaking some ether with H_2O_2 , hydrogen dioxide, and decanting the ether, which contains the real hydrogen dioxide, yielding nascent oxygen.

demonstrator. Note their spectra, and with what ease blood can be detected.

Consult the demonstrator in adjusting the instrument, and if time is given, by means of the comparison prism, compare the spectra of reduced haemoglobin, oxyhaemoglobin and carbonic oxide haemoglobin.

316. Estimation of the Number of Blood Corpuscles by the Haematocrit. Thoroughly cleanse the finger. Prick it near the nail with a sharp needle. Reject the first drop. Suck the second drop into the small graduated capillary tube by means of the little rubber bulb. When the capillary tube is evenly filled with blood, place it, 0° mark out, in the frame and rotate it rapidly for 1 minute. The blood does not coagulate, and the heavy red blood corpuscles gravitate to the distal end of the tube. Next are arranged the white blood corpuscles, and last the clear liquor sanguinis. Each 10 divisions on the tube occupied by the red blood corpuscles correspond approximately to a million red corpuscles per cubic millimetre. Five million corpuscles per cubic millimetre is normal.

LABORATORY QUESTIONS.

1. Which of the three tests for serum albumin was most delicate?
2. Can you invent a method that will detect the alkalinity of freshly drawn blood?
3. To which of the salts detected in the blood will its alkalinity probably be due?
4. Is the coagulation of blood a chemical change?
5. State the part played in the phenomena of coagulation by fibrinogen, paraglobulin and fibrin ferment.
6. Can the approximate age of blood stains be determined by their comparative solubility? Soak in 5 c.c. of cold water a dry blood stain a few days old, one month old, and two months old. What do you conclude?
7. How may the color of cochineal dissolved with alum be distinguished by the spectroscope from a similarly colored blood solution?

MILK.

317. Determination of the Reaction. Note that fresh milk often has a faint alkaline reaction to litmus paper, and sour milk always an acid reaction, due to free lactic acid. Fresh human milk is more alkaline than cow's milk, which may often be neutral or even acid.

318. Microscopical Examination of Milk. Place a drop of milk on a microscope slide. Cover it with a cover glass, and examine the fine emulsion of fat globules.

319. Detection of Lactalbumin. Boil some milk in a beaker. No casein is precipitated, but a scum of coagulated lactalbumin forms on the surface. Skim this off. Wash it, and prove it a proteid by the Xanthoproteic reaction, 257.

320. Separation of Casein. Put some sweet milk in a beaker. Add a little acetic acid, and warm gently. The casein is precipitated. Filter off the whey, and set aside for the next test. Test the casein, and prove it a proteid by 257.

321. Detection of Lactose. Test the whey formed in 320 for lactose by Fehling's test.

322. Detection of Fats. Fill a test tube one-third full of milk. Add 5 c.c. of KOH and a little gasoline. Set the mixture aside and shake frequently. After a few minutes decant, evaporate the gasoline and examine the butter remaining. Butter cannot be dissolved from cow's milk by gasoline or ether without breaking up the emulsive envelope by agitation like churning or alkalies like KOH. Human milk yields its fat directly to ether.

323. Detection of Some Salts in Milk. Evaporate some whey in a porcelain evaporating dish. When nearly dry, heat slowly, to avoid spattering. When dry, raise the heat to red-

ness for a minute, to destroy the organic matter. Cool, and add 5 drops of HNO_3 . Heat gently, dilute with 5 c.c. of water, filter, and test portions as follows:

(1) Add $(\text{NH}_4)_2\text{MoO}_4$, and heat. Yellow ppt. indicates phosphates.

(2) Add AgNO_3 . A slight turbidity indicates chlorides.

(3) Add BaCl_2 . A faint cloud indicates sulphates.

324. Determination of the Specific Gravity. Fill the cylindrical graduate two-thirds full of unskimmed milk. Insert the hydrometer, and take the specific gravity. Remove this instrument, and repeat the determination with the New York Board of Health lactometer. Note that this instrument is so constructed that 100° represents a specific gravity of 1.029, below which unadulterated milk is supposed never to fall.

The laws of New York require milk to have a density of not less than 1.029, and total solids of not less than 12 per cent, of which 3 per cent must be fats.

LABORATORY QUESTIONS.

1. To what cause is the slight alkalinity of milk due?
2. How would boiling affect lactalbumin, globulin and casein present in milk?
3. Acidulate some boiled and some fresh milk, and see if there is evidence that heat has produced a change.
4. How does pasteurized milk differ from boiled or sterilized milk?
5. By the use of rennet or pepsin, how may the so-called sweet pepsin curds for invalids be prepared?
6. Why is lime water often given with milk?
7. How would the addition of water affect the density of milk? The addition of cream?
8. How might the analysis for per cent of total fats and solids be conducted, did time allow? See 322 and 181.
9. How does churning affect milk?

LABORATORY EXERCISES
IN
URINALYSIS

URINE.

APPROXIMATE NORMAL URINARY CONSTITUENTS.

(In 24 hours.)

Water.	1500.	grams,	51 fl. ounces.
Urea.	33.18	"	500 grains.
Uric acid.55	"	8 "
Hippuric acid44	"	6 "
Creatinin.91	"	14 "
Pigments and other organic matter . .	10.	"	154 "
Chlorides of Potassium, Sodium and Ammonium	13.	"	200 "
Sulphates of Sodium and Potassium .	2.20	"	33 "
Phosphates of Sodium and Potassium	3.	"	45 "
Phosphates of Calcium and Magnesium	1.20	"	18 "

EXAMINATION OF NORMAL URINE.

Color.

Urine tints vary from pale yellow to brownish black. The amount of coloring matter is measured by Vogel's scale. Urine is pale yellow after free potations or polyuria from any cause. High-colored urines are induced by pyrexia or abstinence from liquids; orange-red from the presence of santonin in alkaline urine; reddish to brown from blood; brown to black from melonotic cancer or the administration of senna, tannic or carbolic acid, resorcin, naphthalin, etc.; greenish from bile coloring matter.

Odor.

The characteristic urine odor becomes ammoniacal and offensive on standing. Medicinal agents often impart characteristic odors, as asparagus, cubebs, copaiba, garlic, tolu, etc. Turpentine gives a violet odor.

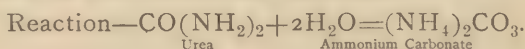
Reaction.

The reaction of mixed urine for 24 hours is normally acid, from the presence of acid sodium phosphate, NaH_2PO_4 . Occasionally urine gives

an amphoteric reaction, changing both litmus colors, due to the presence of acid and alkali salts. Urine may be alkaline after a meal or from administration of alkaline salts, organic acids, etc.

325. Detection of Fixed and Volatile Alkalies. Dip slips of red litmus paper in samples of urine A and B. Both are turned blue. Lay them away to dry. A remains blue and B resumes its former red color. A is alkaline from fixed alkali, Na or K, while B is alkaline from volatile alkali, ammonia.

326. Observation of Urine Changes. Take a sample of fresh urine and place it in a warm place, where it can be examined daily. Test it frequently by litmus paper. Note that it is acid, and that its acidity increases for a few hours, due to acid fermentation, in which mucus acts as a ferment. The acidity then declines, and the urine becomes at last alkaline, from alkaline fermentation, caused by the propagation of fission fungi. The odor becomes ammoniacal as the fungi transform urea into ammonium carbonate.



SPECIFIC GRAVITY.

The normal specific gravity is about 1.020, or, as often written 1020, when taken at 15° C. or 60° F. A decrease in the normal quantity of urine increases its density; an increase in quantity lowers its density. When albumin is present, as in organic albuminuria, or Bright's disease, the density is abnormally low, while in the presence of sugar, as in diabetes mellitus, it is abnormally high.

327. Correction of a Urinometer. Fill a urinometer jar with distilled water at 15° C. (60° F.). In this immerse the urinometer, avoiding contact with the sides of the jar. Read the division on the scale that corresponds with the surface, looking above the meniscus or below, as is found most correct for

the zero reading. Always adhere to this method of reading when using the same urinometer. The urinometer may be several degrees out of the way. If so, note the amount of the variation, and always add or subtract this correction, as the case may be, when taking the specific gravity with the instrument. Many otherwise good urinometers are several degrees at fault. A new instrument should never be brought into use before thus testing its accuracy.

If the urine is warmer than $15^{\circ}\text{C}.$, add 1° to the density for every 4° of extra C. temperature, or for every 7° of extra F. temperature.

TOTAL SOLIDS.

The total solids in urine do not largely vary during health, notwithstanding wide variation in the volume of urine. Solids average 67 grams, or about 1,000 grains, in 24 hours. The total solids are accurately determined by evaporating a measured quantity of urine and weighing the residue, 181. The total solids are determined with sufficient accuracy for clinical purposes from the specific gravity, when the total quantity of urine is known, by the use of Häser's coefficient, 2.33.

328. Determination of Total Solids. Determine the specific gravity of sample C. If the total volume for 24 hours was 1,500 c.c. (51 fl. $\frac{3}{4}$), determine the total solids by the following formula:

$$\frac{\text{Sp. G.} \times .233 \times \text{volume}}{1000} = \text{solids in grams.}$$

UREA.

Urea, $\text{CO}(\text{NH}_2)_2$, forms colorless crystals, odorless, bitter, soluble in water, and acting as either a weak acid or a base. The mean amount in 24 hours' urine is 33 grams, or 512 grains.

329. Preparation of Urea Nitrate. Evaporate a specimen of urine, under the hood, to a syrupy consistency. Cool, and add some pure concentrated HNO_3 , keeping the mixture per-

fectly cool all the time. Cool with ice, if possible, and in a short time rhombic plates of urea nitrate will separate. Urea may easily be obtained from this salt. See any text.

330. The Hypobromite Quantitative Estimation of Urea. Put about 20 c.c. of NaOH in a beaker. Stir, and add bromine, drop by drop, until the solution is orange-yellow. Fill the Doremus ureameter with this solution. Fill the 1 c.c. pipette with urine, and carefully introduce the urine into the long arm of the instrument, taking care to avoid the introduction of air. Decomposition immediately takes place, with the production of nitrogen. After 15 minutes the action is complete. Read off the volume of nitrogen on the graduated scale. The reading gives the weight of urea in 1 c.c. of urine. The total quantity of urea in 24 hours is determined by multiplying this reading by the volume of urine expressed in c.c., or if the decimal point be moved two places to the right, the instrument reads the per cent of urea. Normal urine contains about 0.02 gram per c.c., or 2%.

Reaction— $\text{CO}(\text{NH}_2)_2 + 3\text{NaBrO} = \text{CO}_2 + 3\text{NaBr} + 2\text{H}_2\text{O} + 2\text{N}$.

URIC ACID.

Uric acid, $\text{H}_2(\text{C}_5\text{H}_2\text{N}_4\text{O}_3)$, is insoluble in water. It presents the appearance of red sand. Its neutral salts are soluble in water, but its acid salts only feebly so. For this reason acid urates often ppt. when urine cools. Strong acid, added to a urine highly charged with urates, frequently precipitates uric acid or acid urates in the form of a cloud, that may be mistaken for albumin. Uric acid crystallizes, and may be detected microscopically by its prismatic or whetstone-shaped crystals of a yellowish red color. The normal excretion is about 0.55 grams, or 8 grains, in 24 hours. This quantity is diminished in gout, etc.

331. Separation of Uric Acid. Take 100 c.c. of dense urine in a test tube. Add 10 c.c. of strong HCl. After two days,

decant the liquid from the fine red crystals in the bottom of the tube. Examine these crystals under the microscope.

332. The Murexid Test for Urates. To a little highly colored urine, add a few drops of cold, strong HNO_3 , and evaporate to dryness at a gentle heat. Touch the yellow residue with a drop of NH_4OH . A crimson purple indicates urates.

This color is due to the production of purpurate of ammonia or murexid, a substance which the ancients obtained from shell fish (murex), and used as the royal purple dye.

CHLORIDES.

The chlorides are those of sodium, potassium and ammonium. The mean daily discharge is about 13 grams, or 200 grains, or about 0.5-1%. The amount is diminished in fever. Chlorides may entirely disappear in severe cases of pneumonia. Their reappearance is the first indication of improvement.

333. Detection of Chlorides. Render a test tube of urine acid with a few drops of HNO_3 . Add a strong solution of AgNO_3 (1 \bar{z} to 1 f \bar{z}), drop by drop. If the white ppt. sinks in curdy drops without diffusing, the chlorides are undiminished. If diffusion occurs, chlorides are diminished to about 0.1 per cent. Should no ppt. occur, chlorides are absent.

334. Mohr's Quantitative Estimation of Chlorides. Titrate 10 c.c. of urine, diluted with water, with a standard solution of AgNO_3 ¹, using K_2CrO_4 as an indicator, according to the method 186. From the results calculate the per cent of chlorides.

This method is not quite accurate. Highly colored urines must be diluted to a pale color. 1 c.c. of AgNO_3 is usually allowed for small quantities of interfering substances. The method is more accurate when the 10 c.c. of urine are carefully evaporated, ignited with a little NaNO_3 ,

NOTE 1. This is prepared by dissolving 29.075 grams of AgNO_3 in 1,000 c.c. of water. 1 c.c. of this solution will unite with exactly 0.01 gram of NaCl .

cooled, diluted, neutralized and titrated as above. The most accurate methods are too complicated for use by physicians.

335. Centrifuge Quantitative Estimation of Chlorides. The centrifuge tube is filled to the 10 c.c. mark with filtered urine, acidulated with 2 drops of HNO_3 , and a strong solution of AgNO_3 poured in up to the 15 c.c. mark. The whole is evenly rotated¹ for exactly three minutes, or until the precipitated chlorides are completely packed. The volume of the precipitate is read off. Every $\frac{1}{10}$ c.c. represents 1% of chlorides by bulk. Normal chloride precipitates range from 10-12 % bulk measure.

PHOSPHATES.

Two distinct classes of phosphates exist in the urine: (1) Alkali phosphates of sodium, potassium and ammonium, soluble in water; (2) Earthy phosphates of calcium and magnesium, nearly insoluble in water but soluble in acids. Hence when urine is made alkaline, earthy phosphates precipitate, and when urine undergoes the ammoniacal putrefactive change, earthy phosphates are changed to triple phosphates thus: $2\text{MgHPO}_4 + (\text{NH}_4)_2\text{CO}_3 = \text{H}_2\text{CO}_3 + 2\text{MgNH}_4\text{PO}_4$, or triple phosphate.

336. Detection of Earthy Phosphates. Render a test tube of urine alkaline with NaOH . Heat, and the earthy phosphates are precipitated. Filter the solution, and save the clear filtrate for use in the next experiment.

337. Detection of Alkaline Phosphates. To the clear filtrate from the last experiment, add one-third its volume of magnesium mixture². Alkali phosphates are precipitated in the form of magnesium ammonium phosphate. Examine these

NOTE 1. The centrifuge should be kept well oiled. The arms should be balanced by tubes containing equal quantities of liquids. The machine should not be stopped suddenly nor rotated to excessive speed.

NOTE 2. Magnesium mixture is composed of MgSO_4 and NH_4Cl , each 1 part, distilled water 8 parts, and strong NH_4OH 1 part.

crystals of triple phosphate under the microscope. They will appear in the form of leaves and so-called "coffin lid" crystals, the latter more abundant on standing. These are always present in the microscopical deposit in ammoniacal urine.

338. Centrifuge Quantitative Estimation of Phosphates. Fill the centrifuge percentage tube to the 10 c.c. mark with urine. Add 5 c.c. of magnesium mixture. Both earthy and alkali phosphates are precipitated. Rotate the tube for three minutes and read the percentage. Every $\frac{1}{10}$ c.c. represents 1% of phosphates by bulk. Normal urine yields 8% by volume of phosphate precipitates.

SULPHATES.

The urine contains sulphates of sodium and potassium, in all about 2.2 grams, or 33 grains, in 24 hours. Their determination is of little clinical value.

339. Detection of Sulphates. Render a test tube of urine slightly acid with HCl. Add an excess of BaCl_2 . If a simple turbidity results, sulphates are normal. If an opaque, cream-like precipitate appears, sulphates are excessive. If only a slight cloudiness is produced, sulphates are diminished.

340. Centrifuge Quantitative Estimation of Sulphates. Fill the centrifuge percentage tube to the 10 c.c. mark with urine. Add 2 drops of HCl and then BaCl_2 solution up to the 15 c.c. mark. Rotate the tube three minutes. Read the per cent as usual, counting every $\frac{1}{10}$ c.c. of precipitated BaSO_4 equivalent to 1% of sulphates by bulk. A percentage of 0.8 is normal.

LABORATORY QUESTIONS.

1. On what principle is Vogel's scale constructed?
2. What is the color of urine containing bile?
3. What is the normal reaction of urine, and to what is it due?

4. How do phosphates change in passing from the blood to the urine?
 5. Why will the eating of acid fruits produce an alkaline urine?
 6. Of what value is the detection of volatile alkali in urine?
 7. Can you find a urine with amphoteric reaction?
 8. How long was it before the urine in exercise 326 became alkaline?
- What effect would temperature have on this change?
9. Ammonium carbonate is composed of two gases. Is it volatile?
 10. What is the difference between specific gravity and density?
 11. What is a meniscus, and why so called?
 12. What would cause variations in the reading of a urinometer after its manufacture?
 13. When a urinometer reads 23, what does it mean?
 14. State the formula for determining total solids in the form of a proportion?
 15. How might urea be obtained from urea nitrate?
 16. In what form are proteids chiefly excreted?
 17. Why might urea be called a compound ammonia?
 18. Why is urea an ideal excretory product?
 19. What substance is formed when Br is added to NaOH? Write the reaction.
 20. What is the advantage of knowing the amount of urea excreted?
 21. Name some errors arising in the hypobromite method?
 22. How may urates complicate the test for albumin?
 23. Write the formula for an acid urate.
 24. Will uric acid yield the murexid test?
 25. If time is given, determine the approximate amount of uric acid in urine by precipitating 200 c.c. of urine with 20 c.c. of HCl. Let it stand, decant, wash the crystals, weigh, and calculate the amount in the total twenty-four hours.
 26. Show how the amount of AgNO_3 , 334-Note 1, which it is necessary to make up to 1,000 c.c., can be calculated?
 27. Of what value is a centrifuge to a physician?
 28. What is the value of the determination of chlorides?
 29. What are triple phosphates? What is microcosmic salt?
 30. What is the light, white ppt. that often forms in cold urine?
 31. Write the formulas for two acid and one normal phosphate. Give the reaction of each salt to litmus paper.

32. What urinary deposit will dissolve on warming?
33. A deposit dissolves in a few drops of acid. What is it?
34. A deposit proves insoluble in acetic acid. What is it?
35. What other tests for phosphates might be used?

EXAMINATION OF ABNORMAL URINE.

ALBUMIN.

Among the proteids found in the urine are serum albumin, paraglobulin, mucin, peptone, albumoses, fibrin, haemoglobin, etc. The chief clinical interest centers in serum albumin. It may be present: (1) By degeneration of the renal structure; (2) By a change in the diffusibility of the blood; (3) By an increased blood pressure. The presence of casts or distinctive renal epithelium is the only positive evidence of renal lesions. Urine containing albumin is usually of lowered specific gravity. The quantity of albumin may run as high as 30 grams, or 450 grains, but usually is not over 1-2% by weight. A suitable test for serum albumin *ought* not to react with any other of the above-named proteids, or even coloring matters or salts or medicinal substances present in the urine.

341. Heat and Nitric Acid Test. Take half a test tube of albuminous urine. If alkaline, render it neutral with a drop or two of acetic acid. Bring the upper portion to boiling. Any cloudiness produced is due either to albumin or phosphates. Now add 1 c.c. of strong HNO_3 . Phosphates dissolve. Turbidity due to albumin is intensified.

A large amount of mucin from urinary passages will be mistaken by this test for albumin. When small amounts of albumin are present, exact neutralization is required to insure the detection of acid and alkali albumin. See 274 and 275.

342. Heller's Nitric Acid Test. Place a little clear, strong HNO_3 in a test tube. With a pipette gently float an inch of albuminous urine on the acid surface without mixing. If albumin is present, an opalescent ring appears at the point of contact. With very minute quantities of albumin, the ring appears only after 10 or 15 minutes.

A cloud of amorphous urates sometimes appears, but higher up than the albumin ring, and disappears on gently heating, while the albuminous opalescence is intensified. Any mucin will appear, if at all, near the surface, as it is soluble in strong HNO_3 . This test is one of the most delicate and accurate known, when carefully and intelligently performed.

343. The Ferrocyanide Test. Take half a test tube of clear urine. Mix with it about one-fifth its volume of *fresh*, strong potassium ferrocyanide solution. Now add 10–15 drops of acetic acid. If albumin is present, it will come to view as a white precipitate.

This test precipitates normal acid and alkali albumin, does not act with urates, phosphates, alkaloids or peptones, and the addition of K_4FeCy_6 before the acid keeps mucin in solution. This is one of the most delicate and satisfactory tests. The many other tests for albumin are no better than the three given, and in general are inferior.

344. Esbach's Quantitative Test for Albumin. Fill an Esbach's albuminometer up to the mark U with albuminous urine, then add the picric acid reagent¹ to the mark R. Shake well, cork, and set aside for twenty-four hours. Read off the amount of precipitated albumin on the graduated scale which expresses parts per thousand or grams of dry albumin per liter. Move the decimal point one place to the right, and the reading expresses per cent of dry albumin. This method is approximate only.

345. Centrifuge Quantitative Estimation of Albumin. Fill a centrifuge percentage tube to the 10 c.c. mark with urine. Add 3.5 c.c. of fresh, strong K_4FeCy_6 solution, and 1.5 c.c. of acetic acid. Mix thoroughly, and rotate the tube for three minutes. Read off the amount, and report $\frac{1}{10}\%$ by volume of albumin for each $\frac{1}{10}$ c.c. of precipitate. This is the most accurate method practically available.

NOTE 1. This picric acid reagent contains picric acid 1 part, citric acid 2 parts, and water 50 parts.

GLUCOSE.

Dextrose, laevulose, lactose, etc., are all found in the urine. Dextrose, also termed glucose, or grape sugar, is the sugar of most clinical interest. It exists in the blood normally in small amounts, and in the urine in only the most infinitesimal quantity. *Temporary glycosuria* is caused by fever, cholera, gout and diseases of the lungs, liver, spleen and brain, and by the administration of many toxic drugs. *Persistent glycosuria* may be regarded as symptomatic of grave defect of either the brain, liver, or pancreas. It is the most constant symptom of diabetes mellitus. The presence of sugar usually raises the specific gravity of the urine. The amount may vary from 0 to 1,000 grams, or 0 to 15,000 grains, in 24 hours.

346. Illustration of the Action of the Copper Tests. Take a test tube of CuSO_4 solution. Add KOH , and notice the blue insoluble ppt. of $\text{Cu}(\text{OH})_2$. Divide this among four test tubes. Leave No. 1 as it is. Into No. 2 put a few drops of a solution of glucose, in No. 3 a few drops of glycerine, and in No. 4 a pinch of potassium and sodium tartrate (Rochelle salt). Shake them all. No. 1 is unchanged. Nos. 2, 3 and 4 dissolve to a clear blue solution. Boil them all. No. 1 throws down black CuO , No. 2 a yellow ppt., and Nos. 3 and 4 show no change. Now add glucose to 3 and 4, boil, and both ppt. yellow Cu_2O .

Sugar, glycerine, Rochelle salt, and many other organic compounds keep $\text{Cu}(\text{OH})_2$ in solution. On boiling, glucose and some related sugars, 248, have the power of reducing $\text{Cu}(\text{OH})_2$ to yellowish red Cu_2O . This property is possessed by deteriorated Rochelle salt, and hence all tests using solutions of this salt become in time unreliable.

347. Trommer's Test for Glucose. Take half a test tube of urine. Add 2 drops of dilute CuSO_4 . Next add a liberal quantity of KOH . If sugar is present in any quantity, the blue $\text{Cu}(\text{OH})_2$ first pptd. dissolves to a deep blue liquid, and when boiled ppts. reddish yellow Cu_2O . A precipitated yellow powder is the only certain evidence of sugar. In case

the result seems negative, set aside to settle. If no yellow ppt. appears, glucose was absent. This test is much inferior to the one next described.

348. Fehling's Glycerine Test for Glucose. Prepare some glycerine Fehling's solution thus: Take some CuSO_4 solution in a large test tube. Add a liberal amount of KOH , and then glycerine until when shaken a clear, deep blue solution results¹.

Take half a test tube of this solution, boil, and add 5 drops of urine containing glucose. Again raise it to boiling. A ppt. appears, gradually growing more yellow, and will settle to the bottom, in time, in the form of a reddish yellow powder.

This is the best glucose test for physicians' use. Albumin must be removed if present in any considerable quantity. If 5 drops of urine yield no ppt., add 5 drops more and boil again, continuing until urine equaling in volume the Fehling's solution has been added. Do not add more, as normal urine contains substances which will reduce Fehling's solution, if present in very large amount. A green or blue ppt. in the bottom of the tube is not sugar but precipitated earthy phosphates.

349. Böttger's Bismuth Test for Glucose. First remove all albumin, if present, from some saccharine urine by acidulating the urine slightly, boiling and filtering. In a test tube put equal quantities of urine and KOH . Add a pinch of bismuth subnitrate. Boil, and if glucose is present, the white powder in the bottom of the tube becomes black from the reduction of metallic bismuth. Albumin and all sulphur compounds

NOTE 1. Fehling's glycerine solution may be prepared for office use by the following formula: Dissolve 30 gr. of copper sulphate in $\frac{1}{2}$ ounce of distilled water, add $\frac{1}{2}$ ounce of glycerine, mix thoroughly and add 5 ounces of liquor potassae. Preserve in a rubber stoppered bottle. It keeps indefinitely, and is much superior to Fehling's solution made with Rochelle salt, as this salt, even when kept in a separate solution, deteriorates in a few months.

give the same result, so that this test is much inferior to the previous one.

350. The Phenylhydrazine Test for Glucose. Take 25 c.c. of suspected urine. Add 1 gram of phenylhydrazine hydrochloride (do not allow it to touch the hands), 0.75 gram of sodium acetate and 10 c.c. of distilled water. Keep the solution nearly to the boiling point for an hour. Even minute quantities of sugar yield a yellow ppt., which under the microscope is seen to consist of fine, bright yellow needles of phenylglucosazone, arranged singly or in stars.

This test is one of the most delicate and accurate known, but is too complicated for daily use. It is reliable in the presence of albumin and the products of decomposing urine.

351. The Fermentation Quantitative Test for Glucose. Fill two receivers with suspected urine. Add a small piece of yeast to one, and set both aside for 24 hours. Filter, if necessary, and take the specific gravity of each. The number of degrees of density lost by the fermented urine represents grains of sugar in each fluid ounce. The percentage of sugar may be calculated by multiplying the degrees lost by 0.23. The results are only approximate, and the test is not trustworthy where the quantity of sugar is very small.

352. Fehling's Quantitative Test for Glucose. Take 35 c.c. of Fehling's quantitative copper solution¹ in a beaker. Add twice its volume of distilled water, and heat. Fill a burette with the urine to be examined. When the copper solution boils, slowly drop in the urine, constantly stirring, and boiling until the blue color entirely disappears, which can be best seen by looking through the solution onto a white wall

NOTE 1. The standard solution is prepared as follows: Pure copper sulphate, 4.742 grams; potassium hydroxide, 23.50 grams; strong ammonia, 450 c.c.; glycerine, 38 c.c.; make up with distilled water to 1,000 c.c. This solution keeps perfectly. The color in 35 c.c. of the solution will be exactly destroyed by 0.02 gram of glucose.

or piece of paper. Then read off the number of c.c. of urine used, which will contain exactly 0.02 gram of sugar. The per cent of sugar is computed thus :

$$\frac{100 \times 0.02}{\text{No. of c.c. of urine}} = \text{per cent.}$$

Thus, if the quantity used was 2 c.c.:

$$\frac{100 \times 0.02}{2} = 1 \% \text{ of glucose.}$$

This operation should be conducted as quickly as possible, as the oxygen of the air restores the blue color to the solution, hence the student need not be in a doubt as to the accuracy of the determination if the solution on standing is noticed to be blue.

353. The Polariscope Quantitative Method. This method is very convenient with light-colored urines containing over 1% of sugar. Dark or dense urines must undergo a tedious process of decoloration, and where small amounts of sugar are present the results are inaccurate. If time is at hand, full details of this method may be obtained from the demonstrator, and analyses conducted with the polariscope.

BILE PIGMENTS.

Urine containing bile coloring matters usually has a greenish shade, and a yellow foam forms on shaking. Bile pigments appear in the urine several days before the icteric coloration of the skin is perceptible. They also appear after internal hæmorrhage and phosphorus poisoning.

354. 1. Gmelin's Test for Bile Pigments. Take an inch of strong HNO_3 in a test tube. Drop in one tiny crystal of white sugar and heat. A lemon-colored acid is produced, due to the presence of a little nitrous acid. Carefully overlay this with urine containing bile pigments. A play of colors gradually appears at the zone between the fluids—green, blue, violet, red and yellow. See 302.

2. *Huppert's Modification* of this test is more delicate. To

the suspected urine add lime water and sodium carbonate. A ppt., consisting of lime pigments, falls. Collect and wash this ppt., spread it evenly on a white surface, and touch with a drop of yellow HNO_3 . A trace of bile pigments yields immediately the beautiful colors of Gmelin's reaction.

BLOOD.

Bloody urine is of a reddish to brown color, and always contains albumin. When blood has a renal origin, the color is usually evenly diffused through the urine. In vesical hæmaturia clots are common, and blood appears more abundantly at the close of micturition. The detection of blood casts by the microscope is the most conclusive proof of the renal origin of hæmaturia, common in Bright's disease.

355. The Coagulation Test for Blood. Add 2 drops of acetic acid to the suspected urine, and boil. If the coagulated albumin be tinged red, hæmoglobin is indicated.

356. The Guaiacum Test for Blood. To the suspected urine, rendered slightly acid if alkaline, add 5 drops of fresh tincture of guaiacum. Shake, and add a few drops of hydrogen-dioxide. If blood is present, the solution turns blue. Pus, spermatozoa, etc., yield the same test. See 313.

357. The Microscopical and Spectroscopical Test. Examine a sample of bloody urine with a high power of the microscope, and observe the blood corpuscles. Note that they do not tend to form rouleaux. In old urine their appearance is often so jagged and swollen as to be quite indistinguishable.

The spectroscope may be used to detect blood, as in 315.

PUS.

Urine containing pus always gives the test for albumin. The urine is usually turbid. The microscope will reveal the nucleated pus corpuscles.

358. Donné's Test for Pus. Let the urinary sediment settle. Decant the supernatant urine. Add KOH to the deposit. Pus, if present in any considerable quantity, at once assumes the form of a glairy, gelatinous mass.

ERLICH'S DIAZO REACTION.

This reaction is claimed to be of value in the prognosis of typhoid fever and pulmonary tuberculosis. The reaction is thought to be yielded in these and a few other diseases from the presence of "aromatic substances" produced in the course of the disease.

359. Process. Mix 50 parts of the sulphanilic acid solution¹ and one part of the sodium nitrite solution¹. Add an equal quantity of urine, and render the whole strongly alkaline with ammonia. If the reaction be positive, the solution assumes a carmine red color, which appears on the foam when the solution is shaken. Normal urine yields a yellow color.

LABORATORY QUESTIONS.

1. What proteids are found in the urine? Which, clinically, is the most important?
2. What are some of the causes of albumin in the urine?
3. Which test for albumin in your hands yields the best results?
4. How does the presence of albumin affect the specific gravity of the urine?
5. Why in 341 is it necessary to neutralize the urine with acetic acid?
6. What is the weakness of the heat and nitric acid test for albumin?
7. What precautions must be observed in applying Heller's test?
8. Why should the acid and urine not be mixed in Heller's test?
9. In the ferrocyanide test, what advantage results from adding the K_4FeCy_6 before the acid?

NOTE 1. The sulphanilic acid solution is prepared by dissolving 2 grams of the sulphanilic acid in 50 c.c. of HCl and making up to 1,000 c.c. with distilled water. The sodium nitrite solution is prepared by dissolving 1 gram of sodium nitrite in 200 c.c. of distilled water. These solutions when mixed, liberate diazo-sulpho-benzol.

10. What causes the inaccuracy of Esbach's quantitative test?
11. Would Esbach's test be more accurate if picric acid were replaced by K_4FeCy_6 and acetic acid?
12. Of what clinical value is the detection of albumin?
13. Will Fehling's test detect all the various sugars in the urine?
14. How does the presence of sugar affect the specific gravity of the urine?
15. Write the reaction occurring when KOH is added to $CuSO_4$ solution.
16. Why is it not advisable to use a Fehling's solution made with Rochelle salt?
17. In case one uses Rochelle salt, how can the solution be proved good?
18. What is the greenish precipitate found often in the copper tests for sugar?
19. Why should urine be added to Fehling's solution in small amounts?
20. What are the fallacies of Böttger's bismuth test?
21. Which is the most delicate sugar test?
22. Why is ammonia used in Fehling's quantitative test?
23. Why does Fehling's quantitative solution resume its blue color?
24. What is the highest per cent of sugar yet found in your analyses?
25. How may the nucleus of pus corpuscles be brought into view?
26. What is a pus corpuscle?
27. Report the abnormal constituents and the quantities present, where possible, in bottles D-M, using the printed blank for urinalyses.
28. Will chloroform reduce Fehling's solution? Will chloral? Try it, and note also the chloroform odor evolved.
29. What preserving agent could be added to urine that would not interfere with the tests for sugar and albumin?

LABORATORY EXERCISES
IN THE
TOXICOLOGY
OF THE COMMON
IRRITANT AND NEUROTIC
POISONS

TOXICOLOGY.

CLASS I. IRRITANT POISONS.

Beside the more common poisons of this class mentioned in the brief exercises below, there is a large number of rarer substances which, taken in sufficient quantities, are known to produce violent irritant effects. Among these may be mentioned chlorine, bromine, potassium chlorate, cream of tartar, alum, bismuth subnitrate, salts of barium, gold, silver, platinum, zinc and chromium, castor beans, savine, hellebore, colchicum, gelsemium, cantharides; poisonous mussels, mushrooms, sausage, cheese, etc.

General Symptoms of Irritant Poisons. From a physician's standpoint, poisons are most conveniently classed by their physiological action. This classification is not an inconvenient chemical one, as it likewise broadly divides the poisons into metallic poisons and alkaloids.

The general symptoms of irritant poisons include all phenomena which indicate irritant action upon the mucous lining of the alimentary canal, such as nausea, vomiting, purging, abdominal pains, cramps, etc. Vomited and purged matters are often bloody, and post mortem examinations show more or less gastro-intestinal inflammation, sometimes ulceration, perforation and gangrene, due to secondary processes.

ARSENIC.

360. Microscopical Appearance of Arsenous Oxide. With the microscope examine several different samples of arsenous oxide, As_2O_3 . Note how each differs in the proportions of amorphous powder, broken and perfect octahedral crystals, their size, brilliancy, etc. The source of poison used can often be thus identified.

361. Solubility of Arsenous Oxide. Take four test tubes three-fourths filled with water. Add to each exactly equal quantities of arsenous oxide about the size of a pin's head.

No. 1 dissolves slowly in the cold.

No. 2 dissolves more rapidly when heated.

No. 3 dissolves readily on addition of HCl.

No. 4 dissolves quickly on addition of NaOH.

362. Odor and Taste of Arsenous Oxide. Heat a bit of metallic arsenic on charcoal before the blowpipe. It burns with a bluish white flame, and emits dense white fumes of As_2O_3 , with a garlic odor. Touch a little arsenous oxide for a moment to the tongue, and observe that it is almost without taste.

363. General Reactions of Arsenic. Make a solution of "Rough on Rats" by boiling a pinch with dilute HCl. Using this as an arsenic solution, turn to 141, and confirm all the general reactions of arsenic which are not well in mind.

364. Comparison of Colored Sulphides. Pass H_2S , from the generator in the hood, through four different test tubes containing solutions of arsenic, cadmium, antimony and stannous salts. Notice the colored precipitates in each instance. Confirm the following conclusions by which arsenic and antimony can be separated and identified:

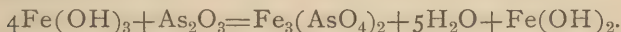
	$(\text{NH}_4)_2\text{S}_2$	NH_4OH	KOH	Cold HCl	Hot HCl
Arsenous Sulphide	Soluble.	Readily sol.	Soluble.	Insoluble.	Slightly sol.
Antimony Sulphide	Soluble.	Insoluble.	Soluble.	Soluble.	Soluble.
Cadmium Sulphide	Insoluble.	Insoluble.	Insoluble.	Soluble.	Soluble.
Stannous Sulphide	Soluble.	Nearly insol.	Insoluble.	Soluble.	Soluble.

365 Action of Antidote—Ferric Hydroxide.

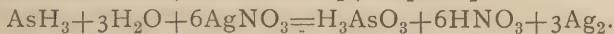
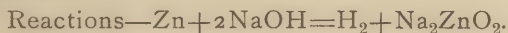
(a) *Preparation.* Precipitate a solution of FeCl_3 with NH_4OH . Strain it through muslin, and collect the moist red ferric hydroxide, $\text{Fe}(\text{OH})_3$. Twelve parts of this is an antidote for 1 part of As_2O_3 . It may be freely administered.

(b) *Demonstration of Efficiency.* Divide a solution of arsenic between two test tubes. Shake the contents of one with the antidote prepared. Filter, and pass H_2S from the generator in the hood through both solutions. In one yellow As_2S_3 is precipitated; in the other no precipitate proves the removal of the arsenic.

Reaction—



366. Rapid Detection of Arsenic.—*Fleitmann's Test.* In a test tube cover a little pure zinc with strong NaOH . Cap the tube with a piece of paper wet with a drop of AgNO_3 solution. Heat nearly to boiling. H is evolved, and, if the materials are pure and arsenic free, the paper remains unstained. Add a little suspected arsenic solution and boil again. If arsenic be present, AsH_3 is evolved, which stains the AgNO_3 spot bluish black. Antimony does not give this reaction.



367. Rapid Examination of Paris Green. Place in the bottom of a clean, dry test tube a little paris green, $\text{Cu}_2(\text{C}_2\text{H}_3\text{O}_2) \cdot \text{CuHAsO}_3$. Have the sides of the tube clean. Heat the bottom very hot. Smell the acetic vapors rising. Observe the white crystals of As_2O_3 sublimed upon the sides of the tube, and the reddish copper in the bottom. Carefully dissolve out some of the As_2O_3 in a little distilled water. Test it by adding a few drops of AgNO_3 , touching it with dilute NH_4OH , and obtaining the yellow precipitate of Ag_3AsO_3 . See 141-3.

368. Examination of Arsenic Stains on Decomposed Tissue. Examine the museum specimens of the mucous mem-

brane from the stomachs of victims of arsenic poisoning. Note in some cases the diffused yellow stains of arsenous sulphide formed by the presence of H_2S in the decaying tissue. Observe in other cases the distinct figured patches of yellowish As_2S_3 formed about undissolved particles of As_2O_3 imbedded in the mucous membrane.

369. Separation of Arsenic from Organic Matter. Place the finely minced tissue containing the poison in a large evaporating dish. Cover it with water. Add $\frac{1}{5}$ its volume of strong HCl . Boil it, and every half minute add a pinch of KClO_3 , with stirring, until the solids all dissolve to a clear yellow liquid. Continue to boil until all the chlorine odor is removed, adding water if it becomes too thick. Strain it through muslin, then through filter paper, and set one-half aside for emergencies. Use the other half for quantitative separation by Marsh's test, as follows: Fit a flask with a thistle tube and a long delivery tube of hard glass drawn out to $\frac{1}{3}$ its diameter at three points. (See sample on the demonstration desk.) Support this on a ring-stand. Place a handful of pure mossy zinc in the generator. Cover the zinc with water, and add moderately strong H_2SO_4 until a slow evolution of hydrogen occurs. Heat the delivery tube to dull redness with a Bunsen burner at a point two inches in front of one of the constricted portions. After the gas has passed for 20 minutes, if no stain appears on the glass the materials may be considered pure. Light the two other burners necessary to heat the tube to dull redness at the two other corresponding points. Slowly drop in the arsenic solution through the thistle tube, and continue the heat and slow evolution of gas until a burner transferred to a new point on the glass no longer deposits a stain, showing that the arsenic has been entirely removed from the solution.

With a triangular file next cut out such portions of the tube as contain arsenic mirrors. Weigh these accurately on a good balance. One of them may be heated, and crystals of As_2O_3 identified by the microscope. Boil them all in a test tube of dilute HNO_3 , and dissolve out the arsenic. Wash the tubes, dry, and weigh them. The difference in weighings gives the weight of metallic arsenic.

The solution in the test tube may be nearly neutralized by NH_4OH , and the arsenic identified by the following tests:

(1) The ammonio-silver test, 141-3.

(2) The sulphide test, 141-1.

LABORATORY QUESTIONS.

1. Write the reaction when As_2O_3 dissolves in water; in KOH .
2. What is white arsenic? Orpiment? Realgar? Ratsbane?
3. Of what value is the microscopical examination of As_2O_3 ?
4. Can you detect arsenic in the sample of green wall-paper furnished?
5. How would you quickly detect Fowler's solution?
6. What is the principal constituent of "rough on rats"?
7. What is paris green? London purple?
8. Why is arsenic such a dangerous poison?
9. What could be used in preparing the As antidote, if ammonia was not at hand?
10. In what form is arsenic dissolved when the tissue is put into solution by chlorine, as in 369?
11. Are eggs, milk or soap useful as antidotes? Try the action of each.

ANTIMONY.

370. Properties of Metallic Antimony. Examine a bit of metallic antimony. Notice that it is extremely brittle, insoluble in alkalies and strong hot HNO_3 , but soluble in a mixture of HCl and HNO_3 . Heated on charcoal before the blowpipe flame, *odorless* white fumes of Sb_2O_3 are evolved (distinction from arsenic).

371. Examination of Tartar Emetic, $\text{KSbO}(\text{C}_4\text{H}_4\text{O}_6)$.

1. It has a nauseous, styptic, acrid taste.
2. It chars when heated, like all tartrates.
3. Heated on charcoal before the blowpipe, it yields brittle metallic beads of Sb, which volatilize to Sb_2O_3 .
4. It is freely soluble in water.
5. Its solutions redden litmus paper.
6. Its solutions are pptd. by HCl , NaOH , Na_2CO_3 , and alcohol.

7. Touched with H_2S or $(\text{NH}_4)_2\text{S}$, it turns orange-yellow from the formation of Sb_2S_3 .

8. A drop of strong solution evaporated spontaneously on a glass slide leaves beautiful rhombic crystals, seen under the microscope.

372. General Reactions of Antimony. Make a solution of tartar emetic. Turn to 142, and review such of the reactions of antimony as are not clearly in mind, noting the distinction from arsenic.

373. Action of Antidotes.

1. *Ferric Hydrate.* Prepare some fresh ferric hydroxide, as in 365 a, and, using a solution of tartar emetic, prove in like manner that $\text{Fe}(\text{OH})_3$ is an antidote for antimony.

2. *Tannin.* To a solution of antimony add a strong solution of tannin. Note the ppt. formed. It may be filtered, and the absence of antimony proved in the filtrate by H_2S .

374. Rapid Detection of Antimony in Solutions. Evaporate a small portion of the suspected solution to a small bulk with two drops of HCl . Pour the concentrated liquid into water. If antimony is present, a white precipitate of antimony oxychloride falls, insoluble on addition of tartaric acid (distinction from bismuth, 139-2).

375. Examination of Antimony Stains on Decomposed Tissue.

Examine the museum specimens of the mucous membrane from the stomachs of victims of antimony poisoning. Note the dark orange stains of Sb_2S_3 , and the golden tinge as the light shines through the tissues. Compare these stains with those of arsenic.

376. Separation of Antimony from Organic Matter. This may be accomplished, and the antimony weighed, by the method used for arsenic, 369. The examination of the antimony mirror is conducted according to the general reactions of antimony, 142.

375. Separation of Both Arsenic and Antimony from Organic Matter. When both arsenic and antimony are found to be present, two principal methods are in use for their separation. The minced tissues are put into solution by HCl and KClO_3 , as under arsenic, 369. Divide the solution into three parts. Put one aside for emergencies. Use $\frac{1}{3}$ for each of the following methods:

METHOD I. Separation by Marsh's Test. Prepare a Marsh's apparatus from a flask, thistle tube and short delivery tube. Start the action, and test the materials as under arsenic, 369. Next pass the gas through a strong solution of AgNO_3 , and feed the suspected fluid, drop by drop, to the generator through the thistle tube. If arsenic or antimony be present, AsH_3 or SbH_3 , or both, are evolved, and form a black precipitate with AgNO_3 . Allow the action to continue until the gas, when passed through a little fresh AgNO_3 , produces no precipitate. (In case any forms, it must, of course, be added to the original AgNO_3 solution.) Filter. The filtrate contains soluble arsenic, and the precipitate consists of Ag and Ag_3Sb .

(a) *Filtrate.* Carefully neutralize the solution with dilute NH_4OH , and a yellow ppt. of Ag_3AsO_3 indicates arsenic, 141-3.

(b) *Precipitate.* Boil the black precipitate with a little HCl and a few drops of HNO_3 . Dilute, and filter off the white AgCl . Antimony in the filtrate is proved by passing H_2S through the solution, which yields the orange-red precipitate of Sb_2S_3 .

METHOD II. *Separation by Hydrogen Sulphide.* Any arsenic present after solution by HCl and KClO_3 is left in the condition of arsenic acid, H_3AsO_4 , which is not readily pptd. by H_2S (see 141-1). Boil it with Na_2SO_3 until the solution smells strongly of SO_2 , to reduce arsenic to the arsenous condition. Boil off the odor of SO_2 . While warm, pass H_2S through the liquid for some time. As_2S_3 and Sb_2S_3 precipitate, with some organic matter by which the color of the sulphides may be modified to a dirty brown. Filter out and wash the sulphides. As they lie on the filter, repeatedly pour over them 15 c.c. of strong NH_4OH . As_2S_3 will dissolve and Sb_2S_3 will remain on the filter. See 364.

(a) *Filtrate.* Evaporate to dryness, mix with K_4FeCy_6 ; insert the mixture in a tube, heat, and metallic arsenic sublimes. Cut the tube, reheat, and identify the octahedral crystals of As_2O_3 with the microscope. These may be dissolved in H_2O and tested:

(1) By the silver-ammonio test, 141-3.

(2) By the hydrogen sulphide test, 141-1.

(b) *Residue.* Dissolve the residue by boiling it with a little concentrated HCl , dilute, filter, if necessary. Pass H_2S through a portion of the solution, and the orange-red Sb_2S_3 falls. Another portion may be concentrated and poured into water, yielding antimony oxychloride, 142-4.

LABORATORY QUESTIONS.

1. What is butter of antimony? Black antimony? Kermes mineral?
2. What is the most common form in which antimony is administered?
3. How may arsenic and antimony sulphides be distinguished by their solubility?
4. Why is tartar emetic incompatible with HCl?
5. Name as many substances as possible incompatible with solutions of antimony.
6. What is the powder of Algoroth?
7. Is $\text{Fe}(\text{OH})_3$ as perfect an antidote for antimony as arsenic? Why?
8. Are eggs, milk and soapsuds antidotes to antimony? Try them, and see.
9. How would you quickly examine a substance, solid or liquid, to see if it contained antimony?
10. How might an antimony mirror be dissolved from a glass tube?

MERCURY.

378. Properties of Corrosive Sublimate. Make a strong solution of mercuric chloride, HgCl_2 . Note its limited solubility, its salty, styptic, powerful taste. The solution reddens blue litmus paper. Save the solution for the following exercises.

379. General Reactions of Mercury. Turn to 137 and 138, and confirm any reactions of mercurous and mercuric compounds that are not fully in mind.

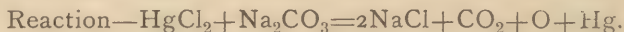
380. Action of Antidotes.

1. *Albumin.* Shake some white of egg in a test tube with HgCl_2 solution. Observe the formation of the insoluble mercury albuminate. Compare this reaction with 268.

2. *Casein.* Shake some milk in a test tube with HgCl_2 solution. The casein is precipitated.

3. *Tannic Acid.* Add a strong solution of tannin to a solution of HgCl_2 , and note the insoluble precipitate formed.

381. Rapid Identification of Mercury Compounds. Mix in a test tube any mercury compound, like calomel or HgCl_2 , with dry Na_2CO_3 . Heat, and a mirror of metallic mercury forms on the tube.



382. Rapid Separation of Bichloride with Ether. Liquids, vomited matters, etc., containing soluble corrosive sublimate may have this salt removed by shaking the material with commercial ether. Decant and evaporate the supernatant ether. Crystals of HgCl_2 will be left nearly pure, which turn scarlet when touched with KI solution. Ether will dissolve about 60 per cent of the corrosive sublimate.

383. Separation of Mercury from Organic Matter.

(1) Boil the minced material, containing HgCl_2 , for some time, with water acidulated with HCl ¹. Strain the liquid off through muslin. Boil the matter again with fresh acid solution. Strain this off and add it to the first extract. Filter this mixture through filter paper and evaporate to one-fourth its volume. Set aside half for emergencies.

(2) Take half the warm solution and insert in it a piece of bright copper foil or wire, which soon becomes covered with a greyish white deposit of metallic mercury, if that metal be present even in minute quantities. This process is known as Reinsch's test, and may be continued until all the mercury has been removed. Put several of these small slips up into a glass tube drawn out like a dropper to a long, fine capillary opening. Heat the tube near the copper, and the mercury will sublime into the capillary neck and form a ring

NOTE 1.—Whenever solutions of mercury have come in contact with albumin or mucous membrane, insoluble mercury compounds have been formed, and considerable boiling is necessary to get the metal again into solution.

of well-defined globules under the microscope. A crystal of iodine pushed up into the cooled tube and left for some hours will slowly volatilize and form HgI_2 , turning the mercury ring scarlet.

LEAD.

384. Examination of Metallic Lead. Heat a mixture of lead acetate and sodium carbonate on charcoal until a small metallic bead is obtained. Hammer it out, and note that it is malleable and will mark on paper. Heat the bead very hot, and notice the yellow incrustation of PbO formed on the charcoal. The metal readily dissolves in HNO_3 .

385. Properties of Lead Acetate. Lead acetate, $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)$, is the most frequent cause of acute lead poisoning. Confirm the following facts:

1. It has a sweet, astringent taste and acetous odor.
2. It crystallizes from strong solutions in 4-sided prisms, seen under the microscope.
3. It dissolves in ordinary water to a milky fluid.
4. A crystal touched with KI turns yellow.
5. A crystal touched with a solution of H_2S turns black.
6. Heated in a test tube, acetous fumes arise. It slowly chars and leaves a reddish brown lead oxide.

386. General Reactions of Lead. Make a solution of lead acetate, turn to 136 and confirm all the reactions for lead that are not clearly in mind.

387. Action of Antidotes. To a solution of lead add the following antidotal reagents:

1. *Magnesium sulphate*, MgSO_4 , ppts. insoluble PbSO_4 .
2. *Albumin* ppts. insoluble lead albuminate.
3. *Soap* ppts. insoluble lead stearate, etc.

388. Separation of Lead from Organic Matter. Boil the minced material with water strongly acidulated with HNO_3 (see Note 1, under Mercury, 383). Strain, filter, nearly neutralize, concentrate, precipitate by H_2S and test the PbS_2 by dissolving it in a little HNO_3 and applying the general tests, 136. If lead is present in any quantity, this will always detect it. If sulphates, as antidotes, have been given, the tissues must be incinerated, the ash dissolved in HNO_3 , and tested.

COPPER.

389. Examination of Verdigris.

(a) *Natural Verdigris.* Examine some of the green compound formed on copper or brass by the action of moisture and the atmosphere. It is $\text{CuO} \cdot \text{CuCO}_3$. Observe that it effervesces with a drop of acid.

(b) *Commercial Verdigris.* Cover a copper cent, or any piece of copper, with acetic acid. Set it aside, and observe the green copper acetate, $\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)$, formed. This resembles commercial verdigris, which is usually copper oxyacetate, $\text{CuO}(\text{C}_2\text{H}_3\text{O}_2)_2$. Using commercial verdigris, confirm the following facts:

1. It is nearly all soluble to a green solution, leaving a little basic residue.
2. It all dissolves on addition of a few drops of HCl .
3. It has an acetous odor, stronger when heated.
4. It has the styptic taste peculiar to copper compounds.

390. General Reactions of Copper. Turn to 140, and, using a solution of copper, confirm the general reactions not familiar.

391. Action of Antidotes. With a solution of copper, confirm the following antidotal reactions:

1. *Albumin* ppts. bluish insoluble copper albuminate.
2. *Potassium Ferrocyanide* ppts. brownish red Cu_2FeCy_6 , insoluble in dilute acids.
3. *Soap* ppts. insoluble blue copper stearate, etc.

392. Rapid Identification of Copper in Solution. Slightly acidulate the solution, which will always be blue or green if copper is present, with HCl. Insert a bright steel needle. Let it remain 5 minutes. Examine the red deposit of copper on it. See how dilute a solution of copper will give this reaction.

393. Examination of Paris Green. The most dangerous ingredient in paris green, $\text{Cu}(\text{C}_2\text{H}_3\text{O}_2) \cdot \text{Cu}(\text{HAsO}_3)$, is arsenic, as seen in 367. The green color is due to copper. Make a solution, add NH_4OH , observe the white precipitate first formed, and later the deep blue solution indicative of copper.

394. Separation of Copper from Organic Matter. The detection of copper presents no difficulty. The material is extracted with acidulated water, strained, filtered, concentrated, precipitated by H_2S , the sulphide dissolved in HNO_3 and tested by the general reactions, 140, or quantitatively by 182.

LABORATORY QUESTIONS.

1. What poison is often eliminated by the saliva? How could it there be detected?
2. How can calomel and corrosive sublimate be distinguished by touching them with KOH?
3. Is calomel soluble in water?
4. Describe a rapid method of detecting mercury, either in a solution or as a solid.
5. What is vermilion? What is amalgam?
6. What is red precipitate? What is cinnabar? What color is it?

7. What other lead salts besides the acetate are used in medicine?
8. What is red lead? White lead? Sugar of lead?
9. What substances are incompatible with lead in solution?
10. Is lead chloride soluble in acids? In cold water? In hot water?
11. Will pure water dissolve lead?
12. Will aerated water dissolve lead?
13. What effect has the presence of organic compounds on the solubility of lead in water?
14. How could canned goods contain lead?
15. What is blue stone?
16. What is Scheele's green and Schweinfurt's green?
17. After what antidotes must emetics be given, and why?

PHOSPHORUS.

395. Properties of Phosphorus. Review experiments 99 and 100 on the forms and solubility of phosphorus, if not clearly in mind.

396. Action of Antidotes.

(a) *Copper Sulphate.* Pour a solution of CuSO_4 on a small piece of waxy phosphorus. Notice after a few moments the coating of insoluble black metallic copper and copper phosphate formed about the phosphorus.

(b) Old turpentine and hydrogen dioxide are advised to oxidize the phosphorus to phosphoric acid, followed by alkalis to neutralize the acid.

397. Detection of Free Phosphorus. *Phosphine Test.* Prepare an active hydrogen generator and light the gas. Note the dim blue flame. Pour in through the thistle tube a solution containing scrapings from a match's head. Take the tube into the dark. Observe the luminous vapors rising in the generator, and that the flame burns green with an outer blue mantle. When the flame is extinguished, the gas has the garlic phosphorus odor.

CARBOLIC ACID, or PHENOL.

398. Properties of Carbolic Acid. Examine carbolic acid, or phenol, C_6H_5OH , and confirm the following facts :

1. It exists as a crystalline solid, which, when warmed with 10% of water, remains a liquid.
2. The clear liquid turns red on long exposure to light. Examine acid which has been so exposed.
3. It has a strong characteristic or "carbolic" odor.
4. It closely resembles creosote. Examine wood creosote.
5. It is not acid, but leaves a greasy stain on blue litmus paper.
6. It dissolves with difficulty when shaken with water.
7. It has a biting taste, and the strong acid raises a white blister on the skin.

399. General Reaction of Carbolic Acid. Turn to 226, and review such general reactions of carbolic acid as are not familiar.

400. Action of Antidotes. To a solution of carbolic acid add the following reagents :

(a) *Saccharate of Lime.* Prepare by pouring water, sweetened with cane sugar, on quicklime, and after standing, decant or filter. A saccharate of lime forms, which increases the amount of lime in solution. Otherwise lime water is as good. A white ppt. falls.

(b) *Soap water* forms a white ppt.

(c) *Magnesium Carbonate* solutions form white ppts.

(d) *Albumin* forms a gelatinous ppt.

401. Detection in Organic Solutions. The characteristic odor readily identifies carbolic acid. It may be readily detected in urine, vomited matters, etc., by shaking with

ether. Decant the supernatant ether, evaporate, and the carbolic acid is left as a minute drop with its characteristic qualities, 226.

OXALIC ACID.

402. Resemblance to Epsom Salt. Oxalic acid, $C_2H_2O_4$, the strongest of the vegetable acids, has often been mistaken for Epsom salt, or magnesium sulphate, $MgSO_4$. Confirm the following differences:

	Oxalic Acid.	Epsom Salt.
Heated . . .	Volatilizes.	Does not volatilize.
Taste . . .	Sour.	Bitter.
Reaction . .	Acid.	Neutral.
NH_4OH . .	Does not ppt. sol.	Ppts. solution.

403. General Reactions of Oxalic Acid. Turn to 213, and confirm any reactions of oxalic acid not clearly in mind.

404. Action of Antidotes.

Alkaline earths.

(a) Prove that $NaOH$, KOH , NH_4OH , Na_2CO_3 , etc., compounds of the alkali metals, neutralize oxalic acid, $H_2C_2O_4$, without precipitation. The soluble oxalates are as poisonous as the acid, hence these alkalies are not antidotes.

(b) Prove that lime water, or milky mixtures of chalk or magnesia, or solutions of magnesium carbonate, will neutralize solutions of oxalic acid, producing insoluble precipitates. Hence these are antidotes.

405. Detection of Oxalic Acid in Organic Matter. Solutions containing much organic material, like vomited matter,

may be tested for oxalic acid by being well digested (water being added, if necessary), the solution strained, filtered, concentrated, and a portion tested by CuSO_4 . Free oxalic acid precipitates bluish CuC_2O_4 , which differs from other copper precipitates in being insoluble in moderately concentrated HNO_3 and HCl . Further concentration will usually allow the acid to crystallize on cooling. When antidotes have been given, the detection is more complicated.

CAUSTIC ACIDS AND ALKALIES.

These substances are so common, their tests so frequently referred to, their neutralization so well understood and their presence in strong solutions used as poisons so readily detected by taste and touch, that they will not be further considered.

LABORATORY QUESTIONS.

1. Why should protective oils not be administered in poisoning by free phosphorus?
2. Would the presence of phosphates in the stomach prove phosphorus poisoning?
3. Why is old turpentine advised as an antidote to phosphorus?
4. Are oxalates in rhubarb poisonous? Why?
5. Would oxalates in the urine prove poisoning by oxalic acid?
6. Why are alkalies not antidotes to oxalic acid?
7. What are antidotes to the mineral acids?
8. What are antidotes to strong alkalies?
9. How could one detect caustic soda?
10. How could one quickly detect sulphuric acid?

CLASS II. NEUROTIC POISONS.

Beside the more common poisons of this class treated of in the following exercises, there are a large number of rarer substances which, administered in larger or smaller doses, produce their toxic neurotic effects. Among these may be mentioned hydrocyanic acid, hyoscyamine, daturine, hyoscine, salanin, lobeline, conine, alcohol, oil of bitter almonds, nitro-benzene, calabar bean, aniline, ptomaines, leucomaines, etc.

General Symptoms of Neurotic Poisons. The general symptoms of neurotic poisons are those arising from specific action on the great nerve centers. In general they are drowsiness, giddiness, headache, delirium, stupor, coma, and sometimes convulsions and paralysis. There are very few antidotes which are chemical in their action.

INTRODUCTORY SEPARATION OF SOME COMMON ALKALOIDS.

CAFFEINE, or THEINE.

406. Sublimation of Caffeine. Put a thin cork ring on a microscope slide, and in it a pinch of dry, finely powdered tea leaves. Cover the ring with another glass slide and warm gently. If the upper glass becomes covered with vapor take it off, fan it until dry, replace it and continue the gentle heat. At about 180° C. a white, filmy deposit will appear on the upper glass. Remove the slide to the microscope and examine the beautiful, distinct needles of theine, or caffeine, $C_8H_{10}N_4O_2$. Powdered coffee may be used instead, but contains a smaller per cent of the alkaloid.

407. Extraction of Caffeine. Add 5 grams of powdered tea to 25 c.c. of water. Boil, digest the solution and filter. Repeat the process with another 25 c.c. of water. Concentrate the united filtrates to 10 c.c., render alkaline with ammonia, and shake with 15 c.c. of chloroform in a large test tube. Separate the chloroform with a small glass pipette. Let it evapo-

rate spontaneously in a watch glass. When examined under the microscope the yellowish white residue may be seen to consist of the characteristic radiating needles of caffeine. Compare the crystals with a commercial sample of caffeine.

408. Sublimation of Theobromine. Sublime a pinch of powdered cocoa bean, as under 406. The needle crystals of caffeine will be observed mixed with the shorter, thicker crystals of theobromine, $C_7H_8N_4O_2$. Compare the form of these crystals with those of commercial theobromine.

409. Extraction of Cinchona Alkaloids. Place about 10 g. of finely pulverized cinchona bark in a beaker. Cover the powder with water, add 10 c.c. of KOH and boil. Pour the solution into a large test tube, and, keeping it hot, shake it several times with a mixture of 15 c.c. of benzol and 5 c.c. of amyl alcohol. This dissolves the alkaloids. Decant the alcoholic layer into another large test tube, add 20 c.c. of distilled water, slightly acidulate with H_2SO_4 and shake repeatedly. The alkaloids, in the form of sulphates, dissolve in the water, which is removed, exactly neutralized with NH_4OH , evaporated one-half, and divided between two watch glasses.

(1) To one add 2 drops of NH_4OH ; a white ppt. falls, composed of all the cinchona alkaloids—quinine, quinoidine, cinchonine, cinchonidine, etc.

(2) Note the bitter taste of the other solution, and after a time beautiful crystals of neutral quinine sulphate $(C_{20}H_{24}N_2O_2)_2H_2SO_4$ form. The other alkaloids remain in solution.

410. Tests for Quinine.

1. *The Fluorescence Test.* Acidulate an alcoholic solution of quinine with dilute H_2SO_4 . A beautiful blue fluorescence appears, destroyed by addition of NaCl.

2. *The Thalleioquin Test.* To a solution of quinine or its salts, add 1 drop of fresh chlorine or bromine water, and render alkaline with 4 drops of ammonia. A deep green color or ppt. is produced. This test is very delicate.

EXERCISES ON THE TOXIC ALKALOIDS.

411. **Extraction of Nicotine.** Boil a large tablespoon full of strong Virginia tobacco with 50 c.c. of water, digest, filter and concentrate the filtrate to 5 c.c. Next cool and add 15 c.c. of strong alcohol. Warm and stir for a few moments, when the gummy resinous matters will be coagulated. The alcohol is to be filtered off, and contains impure nicotine in solution. Evaporate this nearly to dryness. Render slightly alkaline with KOH and shake with 15 c.c. of chloroform in a large test tube. Separate the chloroform with a pipette. Evaporate it in a watch glass. Observe the dark drops of nicotine, $(C_5H_7)_2N_2$, left. Note the odor. Its poisonous properties may be observed by putting two drops far back on the tongue of a small animal.

STRYCHNINE.

412. **Examination of Nux Vomica.** Confirm the following properties which readily identify the commercial powdered nux vomica :

1. It is a light, chocolate-colored, bitter powder.
2. It partially dissolves in water to a bitter solution.
3. Under the microscope the powder seems filled with the broken, fibrous hairs from the seed membrane.
4. It turns deep orange when touched with HNO_3 , 420, *b*.
5. It turns brown when touched with a solution of I in KI.

413. Solubility of Strychnine. Compare the solubilities of strychnine, $C_{21}H_{22}N_2O_2$, and the *salts* of strychnine, by confirming the following table, using a drop of each reagent and a tiny bit of the alkaloid. The same is, in general, true of all alkaloids:

Alkaloid.	Water.	Chloroform.	Ether.	Acids.
Pure Strychnine. Salt of Strychnine.	Nearly insoluble. Soluble.	Soluble. Insoluble.	Soluble. Insoluble.	Soluble. Soluble.

414. General Alkaloid Reactions. Solutions of strychnine salts, and in general all alkaloids, are precipitated by the following reagents. These will be referred to under the other alkaloids. Confirm these reactions, using a drop of the solution on a microscope slide, and touching it with a drop of the reagent.

1. *Alkalies*.—A white amorphous ppt., becoming crystalline.

2. *Mayer's Solution*.¹—A white amorphous ppt., becoming semi-crystalline.

3. *Metatungstic Acid*.²—A white amorphous ppt.

4. *I in KI*.—Reddish brown ppt., partially crystallizing to characteristic crystals.

5. $HgCl_2$.—White amorphous ppt., becoming crystalline.

6. $PtCl_4$.—Pale yellow amorphous ppt., soon becoming crystalline.

7. *Picric Acid in Alcohol Solution*.—Yellow amorphous ppt., becoming crystalline.

8. *Tannic Acid*.—White amorphous ppt.

NOTE 1. Mayer's solution is prepared by adding a solution of KI to a solution of $HgCl_2$ until the precipitate first formed redissolves.

NOTE 2. Metatungstic acid, or phospho-tungstic acid, is prepared by adding H_3PO_4 to a solution of sodium tungstate as long as a precipitate falls and is redissolved.

415. Distinctive Strychnine Tests.

1. *Color Test.* In the bottom of a porcelain dish put two drops of concentrated H_2SO_4 . Add a minute granule of strychnine. It dissolves clear. Near the acid place a tiny crystal of potassium dichromate,¹ $\text{K}_2\text{Cr}_2\text{O}_7$. Next tip the dish so that the acid runs slowly over the crystal. A regular succession of colors occurs, blue, violet, purple, and red fading to pink.

This test may be thus performed if the strychnine is in strong solution. Add a few drops of $\text{K}_2\text{Cr}_2\text{O}_7$, and set aside. Characteristic yellow crystals precipitate. These, when collected and touched with H_2SO_4 , develop the above color reactions, due to definite oxidation compounds.

2. *Crystal Tests.* The crystalline precipitates formed by the general alkaloid reagents are valuable for confirmation. The branching crystals with I in KI, 414-4, are most characteristic.

3. *Physiological Test.* Take an active frog, and with a hypodermatic syringe inject a drop or two of dilute strychnine beneath the skin of its back; $\frac{1}{5000}$ grain will kill a frog in 15-30 minutes. Observe the characteristic tetanic convulsions, which cannot be confounded with anything but tetanus. Note that they occur when the desk is struck or the animal is in any way disturbed.

416. *Detection of Strychnine Tablets.* A portion of the tablet, dissolved in a drop of strong H_2SO_4 and flowed over a tiny crystal of $\text{K}_2\text{Cr}_2\text{O}_7$, develops the characteristic colors directly. In case interfering substances should be present, dissolve a portion of the tablet in 5 c.c. of water, render

NOTE 1. Manganese dioxide, MnO_2 ; lead dioxide, PbO_2 ; potassium permanganate, $\text{K}_2\text{Mn}_2\text{O}_8$; chromic acid, H_2CrO_4 , etc., are advocated.

alkaline with ammonia, add 5 c.c. of chloroform, shake, remove the chloroform, evaporate it drop by drop in one spot on a porcelain dish, cover the spot with a drop of H_2SO_4 , and let the solution flow over a tiny crystal of $\text{K}_2\text{Cr}_2\text{O}_7$, as before. The blue to red colors develop.

417. Detection of Strychnine in Organic Matter. *Roger's and Girdwood's Method.* Digest the comminuted matter with water acidulated with HCl , for some time at a moderate heat. Strain through muslin, and, if possible, through filter paper. If the solution is light colored, and contains little organic matter, leave out the next step, in brackets. [Evaporate the filtrate to dryness, add to the residue strong alcohol, acidulated with a few drops of HCl . Warm, stir, filter, evaporate the filtrate to dryness, dissolve the residue in acidulated distilled water.]¹ Shake this solution with petroleum ether (gasoline), and remove it. Repeat the process as long as the gasoline is colored. When the coloring matters are thus removed, render the aqueous solution alkaline with ammonia, and shake with chloroform twice, each time withdrawing the *clear* chloroform. Evaporate this chloroform, in a few spots, on porcelain. Dissolve the residue in a drop of concentrated H_2SO_4 . If the spot blackens, warm it until the organic matter chars, dissolve it in a little water, render alkaline, and extract with chloroform as before. Continue this until the residue dissolves clear in H_2SO_4 , and then apply the color test with $\text{K}_2\text{Cr}_2\text{O}_7$, 415-1, and crystal tests, 415-2. When much decayed organic matter is present it is often impossible to get a color reaction. The extract still is bitter, and answers the physiological test.

NOTE 1. This step removes much organic matter.

BRUCINE.

418. Solubility of Brucine. Observe that brucine, $C_{23}H_{26}N_2O_4$, resembles strychnine in solubility, but dissolves more readily in water and alcohol.

419. General Alkaloid Reactions. The general reagents, 414, precipitate brucine, but less readily than strychnine.

420. Distinctive Brucine Tests.

1. Color Tests.

(a) H_2SO_4 dissolves brucine to a faint rose-red solution, which does not yield the strychnine play of colors with $K_2Cr_2O_7$.

(b) HNO_3 dissolves brucine to a deep red solution, passing rapidly to yellow when heated. This, cautiously touched with a drop of stannous chloride solution, $SnCl_2$, develops a purple color.

2. *Physiological Test.* Brucine acts on frogs like strychnine, 415-3, but less energetically.

421. Detection in Organic Matter. Brucine may be detected by Roger's and Girdwood's method, 417.

LABORATORY QUESTIONS.

1. To what are the poisonous qualities of plants due?
2. Why are alkaloids so called?
3. What are the peculiarities of the volatile alkaloids?
4. Which alkaloid so far examined is most highly nitrogenized?
5. How does the roasting of coffee affect its strength?
6. What alkaloid does the kola-nut contain?
7. Is there any relation between theobromine and uric acid?
8. In what forms do alkaloids usually occur in plants?
9. What is nux vomica, and what are its alkaloids?
10. Why does nux vomica turn orange-red when touched with HNO_3 ?
11. Are alkaloids soluble in water? In ether and chloroform?

12. How can alkaloids be rendered soluble in water?
13. In what chemical form are alkaloids usually administered?
14. How could one detect a strychnine tablet in 15 seconds?
15. Outline the general method of separating alkaloids.
16. Describe the method of Dragendorff.
17. Describe the method of Stas and Otto.
18. How do strychnine colors, 415-1, resemble bile colors, 302?
19. What is the difference between the sulphate and bisulphate of quinine?

MORPHINE.

422. Properties of Opium. Examine a piece of opium and observe:

- (1) Its bitter, blistering taste.
- (2) Its characteristic odor, clinging even to its solutions.
- (3) Its gummy consistency, or when dry, hard and brittle, of a reddish brown color, often mixed with small pieces of skin from the poppy pod.

(4) Aqueous infusions of opium turn red on addition of neutral FeCl_3 , due to the formation of ferric meconate. The color is not destroyed by a solution of corrosive sublimate. This test is quite conclusive of opium in solution.

423. Extraction of Morphine. Powder a piece of opium as large as a bean. Boil it with 30 c.c. of water, filter, and evaporate the filtrate to 5 c.c. Pour this into a mixture of 25 c.c. of ether and 10 c.c. of alcohol rendered alkaline with strong ammonia. Shake the mixture and set aside for some hours. The ethereal solution dissolves part of the alkaloid, and the remainder, as impure crystals of morphine, may be seen in the bottom. Remove a few drops of liquid containing some crystals from the bottom by means of a pipette. Examine these under the high power of the microscope, and observe their irregular quadrilateral and rounded forms.

Put some of these tiny crystals on glass and set aside to dry. When the following tests are familiar, identify these crystals as morphine, $C_{17}H_{19}NO_3$, by the general and special reagents.

Morphine crystallizes with difficulty from solutions containing organic matter. With care very good results can be obtained by this process.

424. General Alkaloid Reactions. Apply a drop of each reagent in 414 to a drop of strong solution of morphine hydro-sulphate, and note the precipitation.

425. Distinctive Morphine Reactions. There is no *one* reaction which is absolutely distinctive of morphine. There is, however, no *crystalline* substance but morphine that answers the four following tests:

1. *The Sulpho-molybdic Test.* Dissolve in two drops of H_2SO_4 a crystal of ammonium molybdate. Molybdic acid is liberated. A speck of solid morphine touched with this immediately dissolves to a momentary crimson-purple color, which in ten minutes becomes beautiful deep blue, and gradually fades. When organic matter is present the pink hue is obscured, but the deep blue color usually appears later.

2. *Nitric Acid Test.* A drop of HNO_3 strikes with a little morphine a red color, fading to yellow. When this is touched with a drop of $SnCl_2$ solution, no purple hue results (distinction from brucine).

3. *Ferric Chloride Test.* A crystal of morphine, or a drop of a concentrated solution, strikes a deep greenish blue with a drop of $FeCl_3$, discharged by free acids, alkalies and heat. HNO_3 added turns the color orange.

4. *The Iodic Acid Test.* A drop of moderately strong morphine solution turns brown when treated with iodic acid,

due to the liberation of free iodine. A little starch paste first mixed with the iodic acid remains colorless, but turns blue when added to morphine. This is a delicate test.

426. Detection of Morphine Tablets. Usually a bit of a morphine tablet touched with a drop of sulpho-molybdic solution, 425-1, turns red, and becomes finally deep blue. This is a sufficient test.

When interfering substances are present, dissolve the tablet in 5 c.c. of water. Render alkaline with ammonia, shake with an equal volume of *hot* amyl alcohol (or a mixture of ether 3 parts and alcohol 1 part). This will dissolve the morphine. Separate the supernatant liquid and let it evaporate on several spots. To these apply the sulpho-molybdic test, 425-1, and others, if desired.

427. Detection of Morphine in Organic Matter. The organic matter is digested with water, acidulated with acetic acid, at a temperature never exceeding 70° C. (160° F.), strained and filtered. The process is repeated, the filtrates are united, and concentrated to a small bulk by slow evaporation, at a temperature never above 70° C. The solution is then shaken with petroleum ether (gasoline), and the supernatant fluid removed as long as it removes coloring matters. The solution is then once shaken with amylic alcohol to remove further coloring matter. Next render the solution alkaline, shake with twice its volume of *hot* amylic alcohol. The amyl alcohol is removed and the process repeated. The amyl alcohol, spontaneously evaporated in a watch glass, often leaves crystals. When much organic material was present crystals can rarely be obtained. Test specks of the solid residue by 425-1, 2, 3, 4. Dissolve the remainder in very dilute acetic acid, and test a drop by I in KI,

414-4, which ppts. yellowish brown amorphous granules, becoming, after a time, branching, moss-like crystals, under the microscope.

528. Reactions of Apomorphine. Prepare a little solution of apomorphine, $C_{17}H_{17}NO_2$, in HCl, and confirm the following reactions:

1. *The Iron Test.* A drop of $FeCl_3$ yields an amethyst color.

2. *The Nitric Acid Test.* Concentrated HNO_3 yields a red color.

3. *The Manganese Test.* A few grains of MnO_2 yields a green color, turned reddish brown by a crystal of oxalic acid.

ATROPINE.

429. General Alkaloid Reactions. Examine atropine or its salts. Note its bitter taste, and its solubility in drops of water and chloroform, like other alkaloids. Observe that the general alkaloid reagents, 414, precipitate atropine, $C_{17}H_{23}NO_3$.

430. Distinctive Atropine Tests.

1. *Vitali's Test.* Touch a bit of atropine with a drop of fuming HNO_3 . Evaporate to dryness and touch the residue with a drop of a solution of KOH in absolute alcohol. A violet color is produced.

2. *Wormley's Test.* Saturate a solution of HBr with bromine. With a drop of this precipitate a drop of an atropine solution on a microscope slide. After a time the microscope will reveal characteristic crystalline leaf clusters.

3. *Sulphuric Acid Test.* A little atropine dissolved in strong H_2SO_4 and heated, gives off a fragrant odor of roses.

4. *The Physiological Test.* Place in a cat's eye one drop of a very dilute solution of atropine. The pupil, in the course of a few minutes, widely dilates. Dilation produced by atropine is more persistent than that produced by any other alkaloid.

431. Detection of Atropine Tablets. The sugar in tablets interferes with some direct tests.

The physiological test with a drop of the solution in a cat's eye is a quick and convenient method.

The color reaction is easily obtained by dissolving the tablet in 5 c.c. of water, rendering alkaline with ammonia, shaking with 5 c.c. of chloroform, evaporating the chloroform on a few spots and applying Vitali's test (430-1) and Wormley's test (430-2).

432. Detection of Atropine in Organic Matter. The method of separating atropine from organic matter in general is that outlined under strychnine. The temperature should never be high, and the residue from the chloroform cannot be heated with H_2SO_4 and so purified. Where large amounts of putrid matter are present, a crystalline product often cannot be obtained. The solution, however, is bitter, and dilates the pupil. Other members of the belladonna family, it must be remembered, also have this power.

COCAINE.

433. General Alkaloid Reactions. Prepare a small amount of a solution of cocaine hydrochloride, $\text{C}_{17}\text{H}_{21}\text{NO}_4\text{HCl}$. Observe the precipitates with the general alkaloid reagents, 414. Especially notice the microscopical appearance of the characteristic stellate crystals formed with PtCl_4 and with picric acid.

434. Distinctive Cocaine Tests.

1. *Vitali's Test.* Add to a bit of cocaine a drop of fuming HNO_3 , evaporate it to dryness, touch with a drop of alcoholic potash and note the odor of peppermint.

2. *Physiological Test.* A drop of cocaine solution, as strong as 4%, on the tongue produces numbness.

LABORATORY QUESTIONS.

1. What alkaloids are extracted from opium?
2. Why is opium bitter?
3. What alkaloid studied is the most difficult to separate?
4. What relation does apomorphine bear to morphine?
5. What alkaloids are related to atropine? Do they give Vitali's test?
6. Give a rapid method of detecting atropine?
7. Why is morphine not extracted with chloroform or ether?
8. What is the source of atropine? Of cocaine?
9. Is morphine soluble in water? In alcohol?
10. How did morphine receive its name?
11. What is meconic acid, and where does it occur?
12. By what simple test can laudanum be identified?
13. What alkaloids does cinchona bark contain?
14. In what ways do ptomaines and leucomaines resemble alkaloids?

TABLE V.

**A Guide in the Rapid Examination of Suspected Matters
for Common Poisons.**

A thorough examination of the material should be made. Odors may detect volatile poisons ; a styptic taste, metallic irritants ; a bitter taste, alkaloids ; a sour taste, acids ; a biting taste, alkalies. Solids should be examined with the microscope, any seeds, berries, pods, hairs, wing-cases, etc., carefully noted. This investigation, with some characteristic symptoms, appearances, etc., will nearly always direct to some one of the following groups :

A. Volatile Poisons.

An acidulated portion heated evolves the odor of

1. *Cyanides*. Confirm by 129-3-b.
2. *Carbolic Acid*. Confirm by 401 and 226.
3. *Chloroform*. Confirm by 201-b.
4. *Phosphorus*. Confirm by 397.

B. Acids and Alkalies.

The substance has an acid or alkaline reaction. Test for

1. *Acids*. H_2SO_4 , 81 and 79 ; HNO_3 , 98 ; HCl , 43 ; H_3PO_4 , 106 ; $\text{H}_2\text{C}_2\text{O}_4$, 213 and 405.
2. *Alkalies*. KOH , 170 ; NaOH , 171 ; NH_4OH , 172 and odor.

C. Metallic Irritants.

Acidulate a portion with HCl and boil with a piece of bright copper.

1. *A Grayish Deposit—Arsenic*. Confirm by 141-5 and 366.
2. *A Bluish Deposit—Antimony*. Confirm by 374 and 142-1.
3. *A Bright Deposit when Rubbed—Mercury*. Confirm by 383-2, 381 or 382.

A needle dipped in the acid solution receives a reddish deposit and the solution is green or blue.

4. *Copper*. Confirm by 140-2.

If none of these reactions occur and H_2S gives a black ppt., it may be

5. *Lead*. Confirm by 136-2 and 5 (see 388).

D. Alkaloids.

A filtered portion of the solution, acid from HCl , is shaken with ether to remove fats and coloring matters, if necessary, rendered alkaline with ammonia, shaken with chloroform, and the chloroform evaporated on several spots. A residue remains.

1. *Strychnine*. Confirm by 415-1.

2. *Atropine*. Confirm by 430-1. Dissolve in acidulated water and test by 430-4.

Another portion shaken with hot amyl alcohol and evaporated on several spots with gentle heat.

3. *Morphine*. Confirm by 425-1 and 4.

NOTE. These methods are not searching enough for a legal trial, but are useful for clearing up a diagnosis before it is too late.

TABLE OF SYMBOLS, VALENCIES AND ATOMIC WEIGHTS.

Element.	Symbol.	Valency.	Atomic Weight. U. S. P.	Element.	Symbol.	Valency.	Atomic Weight. U. S. P.
Aluminum	Al	IV	27.04	Molybdenum . .	Mo	II IV	95.9
Antimony	Sb	III V	119.06	Nickel	Ni	II	58.6
Argon	A		40.7	Niobium	Nb	V	93.7
Arsenum	As	III V	74.9	Nitrogen	N	III V	14.01
Barium	Ba	II	136.9	Osmium	Os	VI	193.
Beryllium	Be	II	9.03	Oxygen	O	II	15.96
Bismuth	Bi	III V	208.9	Palladium	Pd	II IV	136.35
Boron	B	III	10.9	Phosphorus . . .	P	III V	30.96
Bromine	Br	I	79.76	Platinum	Pt	IV	194.3
Cadmium	Cd	II	111.5	Potassium	K	I	39.03
Cesium	Cs	I	132.7	Rhodium	Rh	II IV	102.9
Calcium	Ca	II	39.91	Rubidium	Rb	I	85.2
Carbon	C	IV	11.97	Ruthenium	Ru	II IV	101.4
Cerium	Ce	II IV	139.9	Samarium	Sm	III	149.62
Chlorine	Cl	I	35.37	Scandium	Sc	III	43.97
Chromium	Cr	IV	52.	Selenium	Se	II IV VI	78.87
Cobalt	Co	II	58.6	Silicon	Si	IV	28.3
Copper	Cu	II	63.18	Silver	Ag	I	107.66
Didymium	Di	III V	142.	Sodium	Na	I	23.
Erbium	Er	III	166.	Strontium	Sr	II	87.3
Fluorine	F	I	19.	Sulphur	S	II IV VI	31.98
Gallium	Ga	III	69.9	Tantalum	Ta	III V	182.
Germanium	Ge	II IV	72.3	Tellurium	Te	II IV VI	125.
Gold	Au	I III	196.7	Terbium	Tr		159.1
Hydrogen	H	I	1.	Thallium	Tl	I III	203.7
Indium	In	III	113.6	Thorium	Th	II IV	231.9
Iodine	I	I	126.53	Tin	Sn	II IV	118.8
Iridium	Ir	II IV	192.5	Titanium	Ti	IV	48.
Iron	Fe	II IV	55.88	Tungsten	W	IV	183.6
Lanthanum	La	III	138.2	Uranium	U	II	238.8
Lead	Pb	II	206.4	Vanadium	V	III V	51.1
Lithium	Li	I	7.01	Ytterbium	Yb		172.6
Magnesium	Mg	II	24.3	Yttrium	Y	III	88.9
Manganese	Mn	II IV	54.8	Zinc	Zn	II	65.1
Mercury	Hg	II	199.98	Zirconium	Zr	IV	90.4

TABLE OF EQUIVALENT WEIGHTS AND MEASURES.

APOTHECARIES' FLUID MEASURE.

1 minim		η	=	.062 c.c.
60 minims	= 1 fluid drachm,	$\mathfrak{f}\mathfrak{z}$	=	3.697 c.c.
8 fluid drachms	= 1 fluid ounce,	$\mathfrak{f}\mathfrak{z}$	=	29.573 c.c.
16 ounces	= 1 pint,	O	=	473.179 c.c.
8 pints	= 1 gallon,	Cong.	=	3.785 l.

APOTHECARIES' WEIGHT MEASURE.

1 grain		gr.	=	.0648 grams.
20 grains	= 1 scruple,	\mathfrak{D}	=	1.296 grams.
3 scruples	= 1 drachm,	\mathfrak{z}	=	3.888 grams.
8 drachms	= 1 ounce,	\mathfrak{z}	=	31.104 grams.
12 ounces	= 1 pound,	\mathfrak{lb}	=	373.242 grams.

ENGLISH LINEAR MEASURE.

1 inch	=	2.54 centimeters.
12 inches	= 1 foot	= 30.48 centimeters.
3 feet	= 1 yard	= 91.44 centimeters.

METRIC WEIGHT MEASURE.

1 milligram		m.g.	=	.01543 grains.
10 milligrams	= 1 centigram,	c.g.	=	.1543 grains.
10 centigrams	= 1 decigram,	d.g.	=	1.543 grains.
10 decigrams	= 1 gram,	g.	=	15.43 grains.

METRIC FLUID MEASURE.

1 cubic centimeter, c.c.	= 16.231 minims	= .061028 cubic inches.
1,000 c.c.	= 1 liter, l.,	= 33.815 fluid ounces = 61.028 cubic inches.
1 c.c. of distilled water	at 4° C.	weighs 1 gram.

METRIC LINEAR MEASURE.

1 millimeter,		m.m.	=	.03937 inches.
10 millimeters	= 1 centimeter,	c.m.	=	.3937 inches.
10 centimeters	= 1 decimeter,	d.m.	=	3.937 inches.
10 decimeters	= 1 meter,	m.	=	39.37 inches.

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